Application of Electron Microscopy Techniques to the Investigation of Space Shuttle Columbia Accident

SEMS 2005

By

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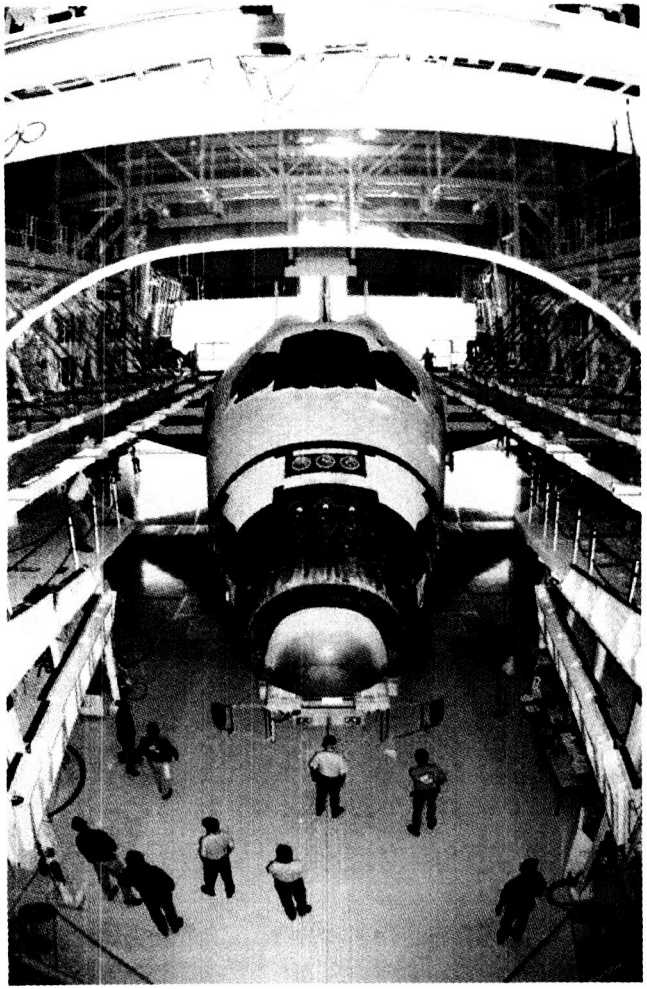
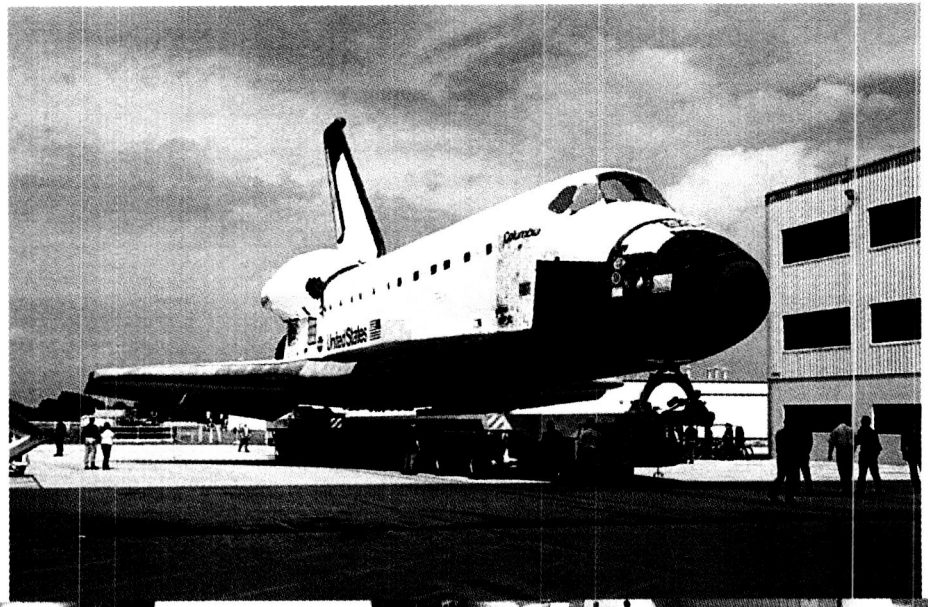
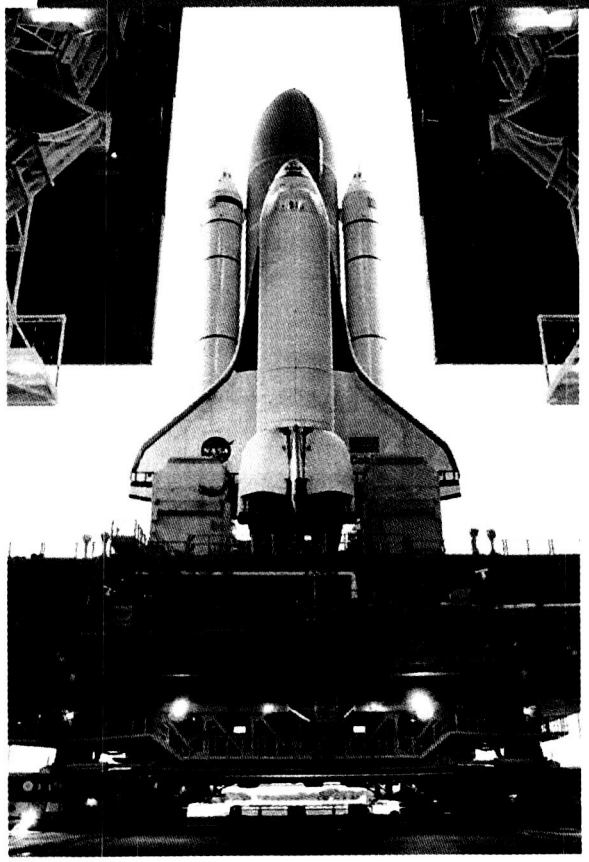
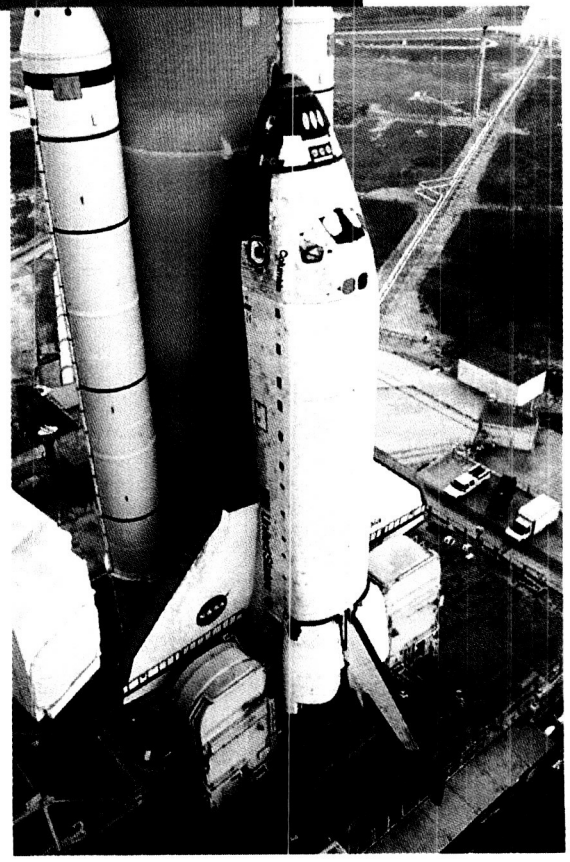
Acknowledgement: This work is a result of a team effort involving KSC, JSC, MSFC, LaRC, GRC, Boeing, USA and Columbia Accident Investigation Board personnel.

IN MEMORY
OF STS-107
ASTRONAUTS

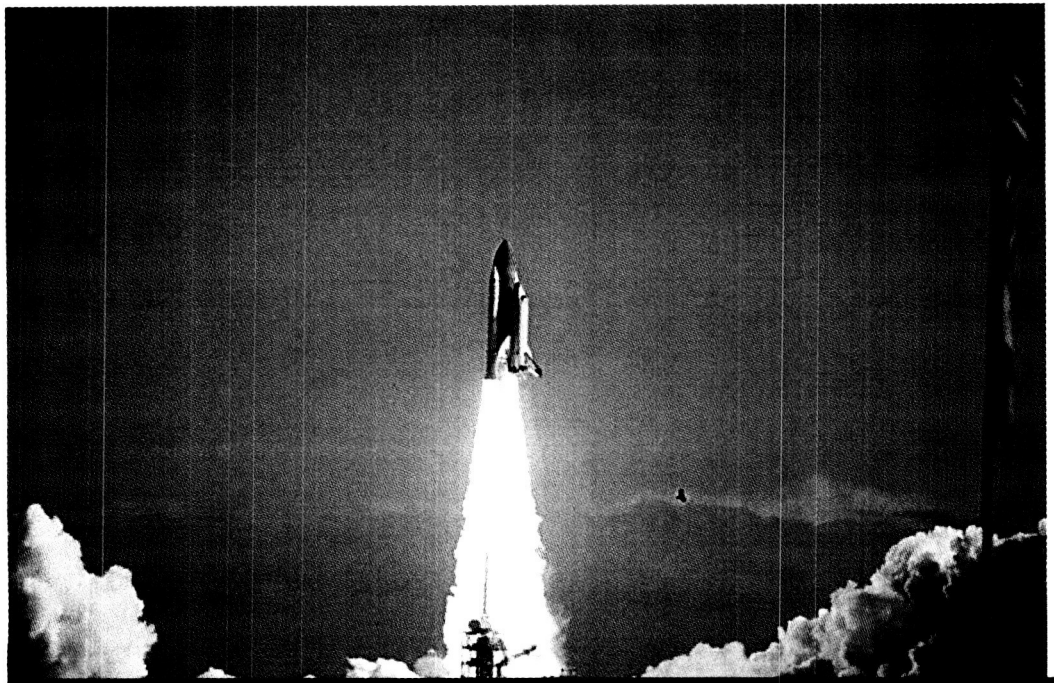


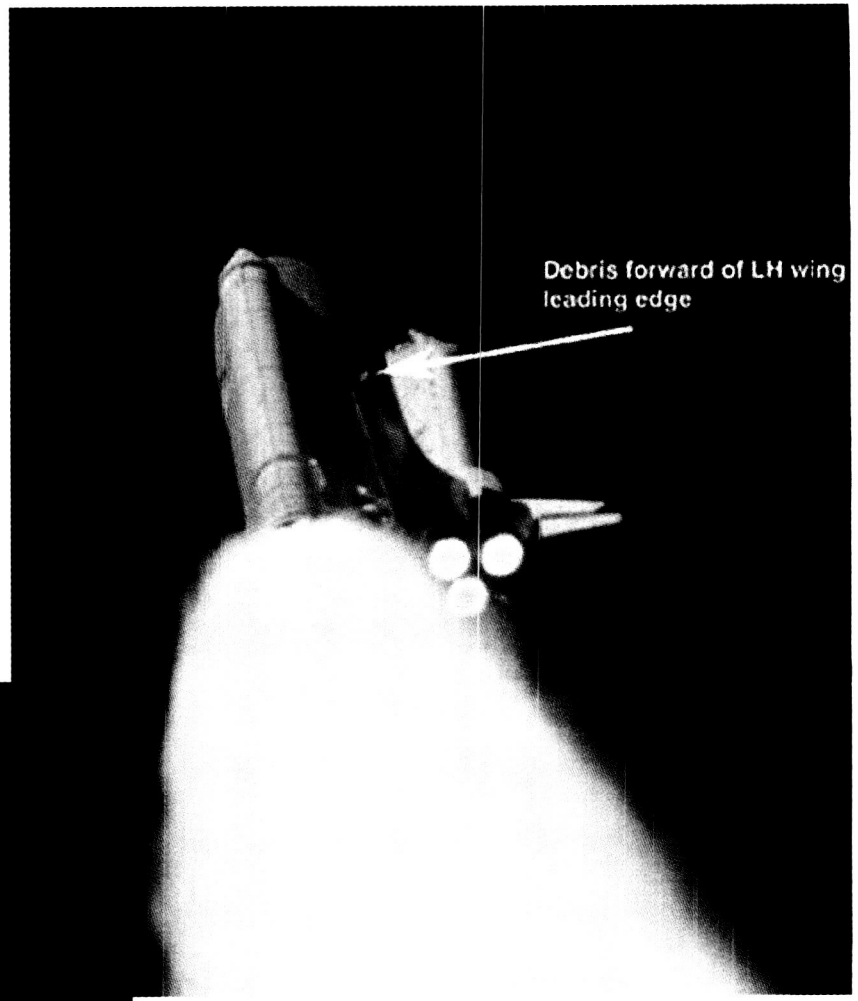




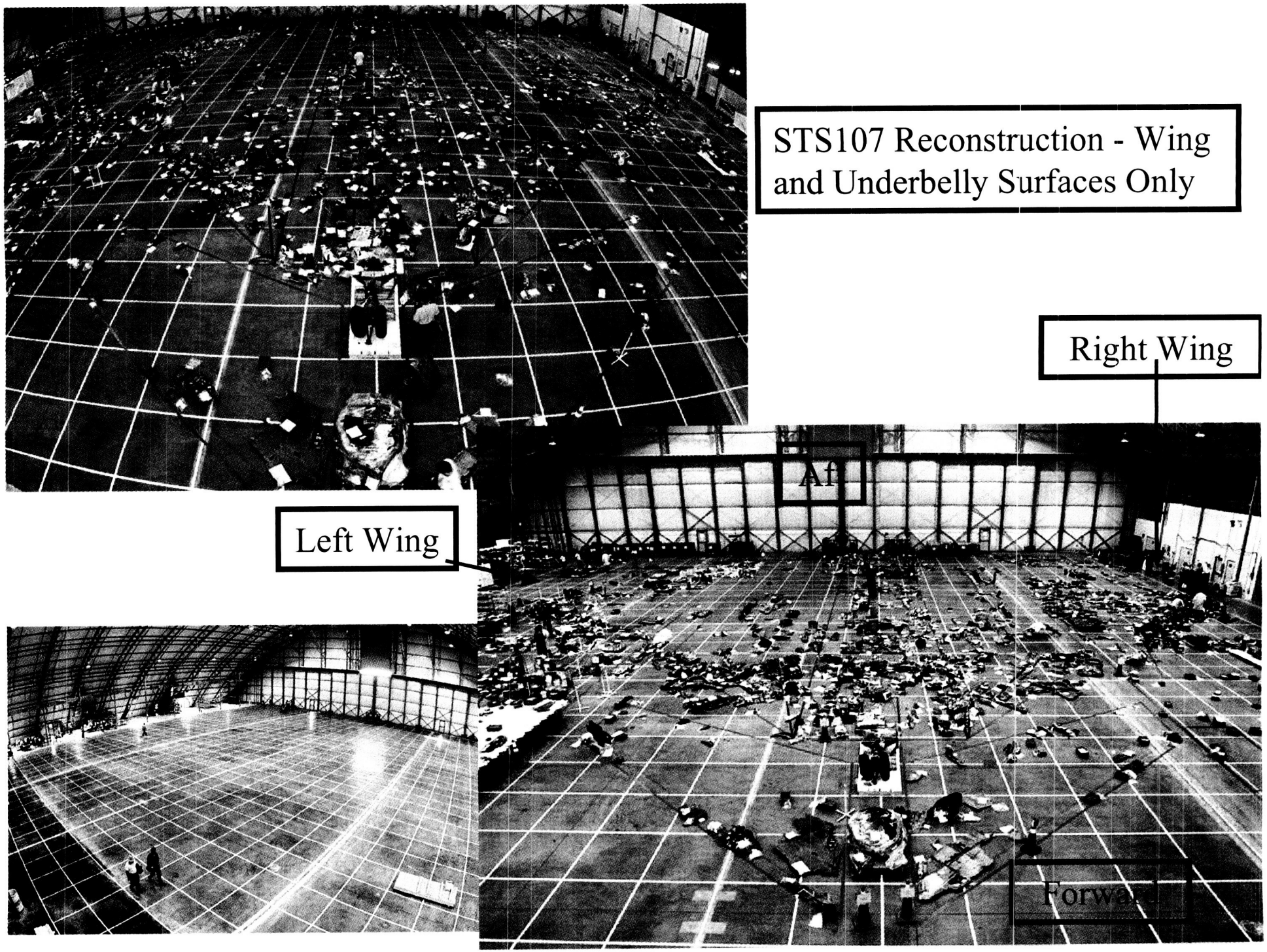


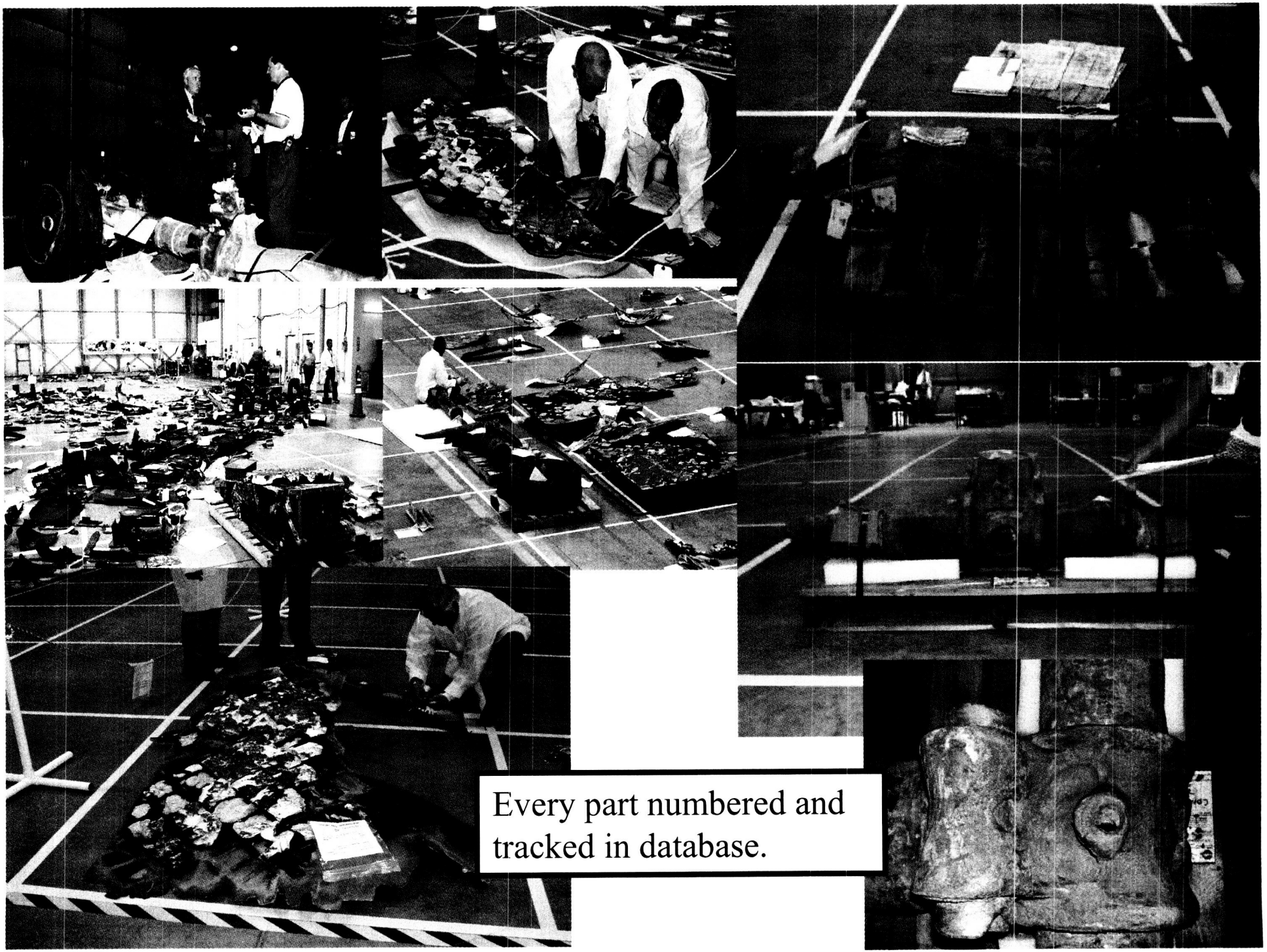
Space Shuttle Columbia
Roll Out



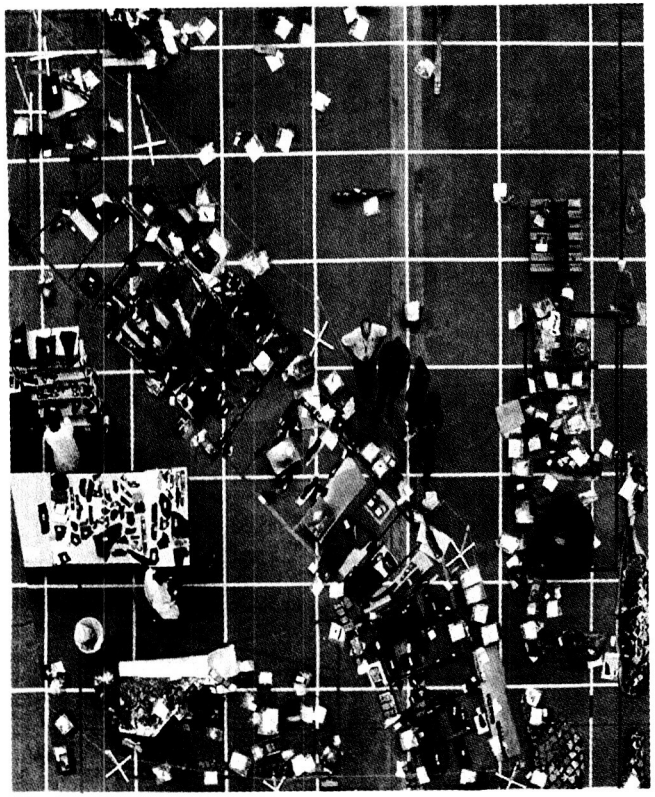


STS107 Launch

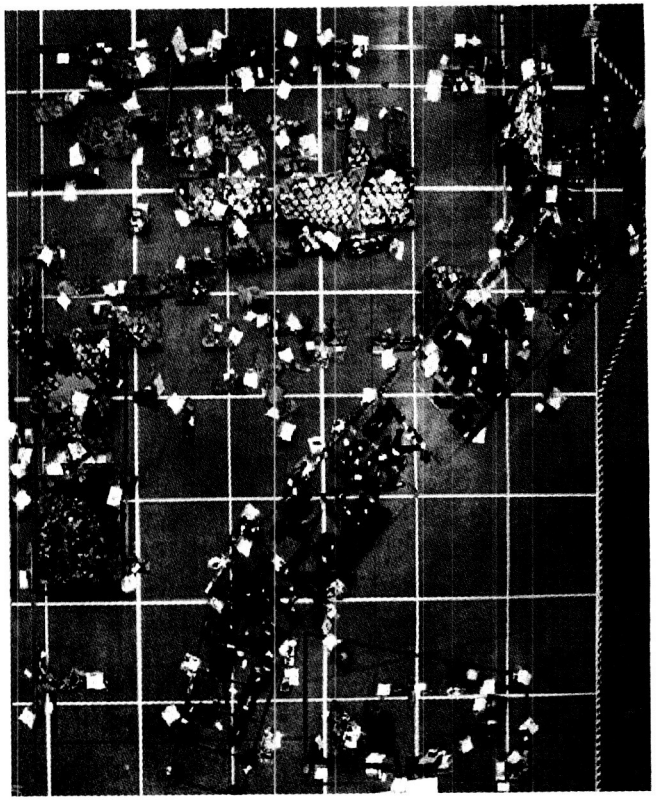

STS107 Reconstruction - Wing and Underbelly Surfaces Only
Right Wing
Aft
Left Wing
Forward



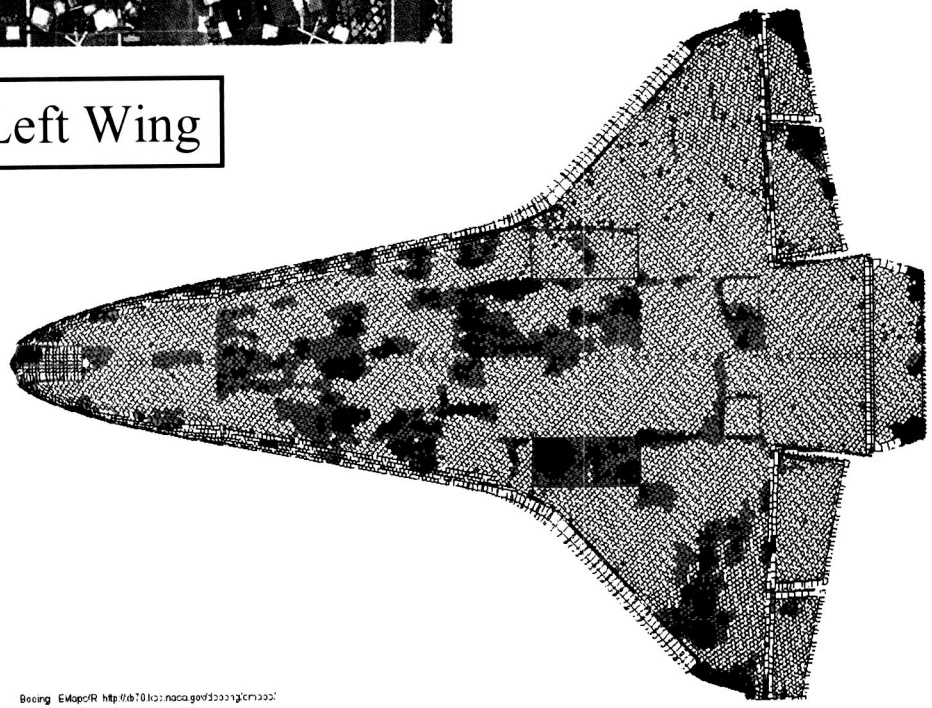
Every part numbered and tracked in database.

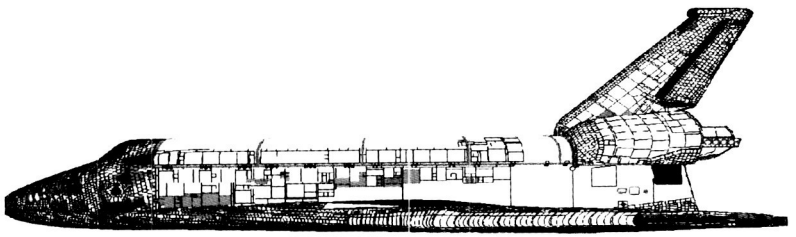


Left Wing



Right Wing





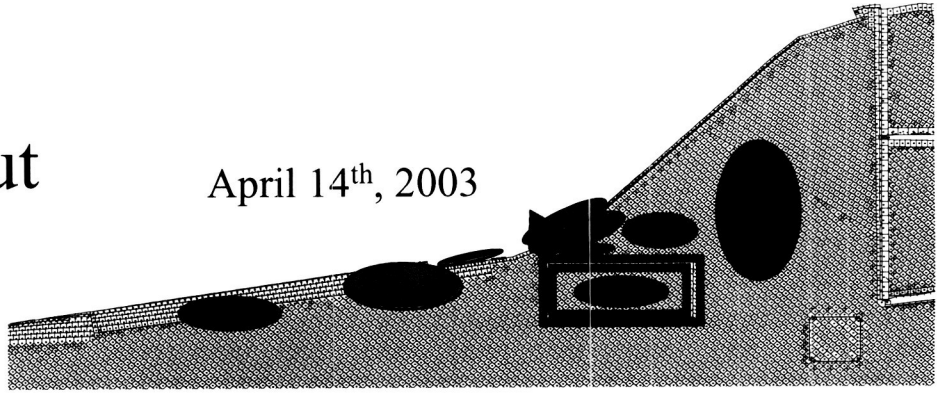
Failure Analysis Challenges

- Where to Begin? What Questions to ask?
- How many parts to analyze?
- Priority of each Failure Analysis?
- Distinguish between damage in flight vs free fall and impact.

M&P Team to provide Factual data about materials condition.

Failure Analysis Locations

- All indications of Left Wing Failure. Where??
- Initial Analysis focused on multiple Left Wing parts:
  - Midbody Panel
  - Main Landing Gear Strut
  - Uplock Rollers
  - Tires
  - Leading Edge Carrier Panel Fastners
  - Tiles
  - Leading edge RCC material deposit



Key Finding

- Data Recorder Found–"black box".
- Black box showed increase in temperature in left wing leading edge prior to any other sensor failure indicating a possible breach.

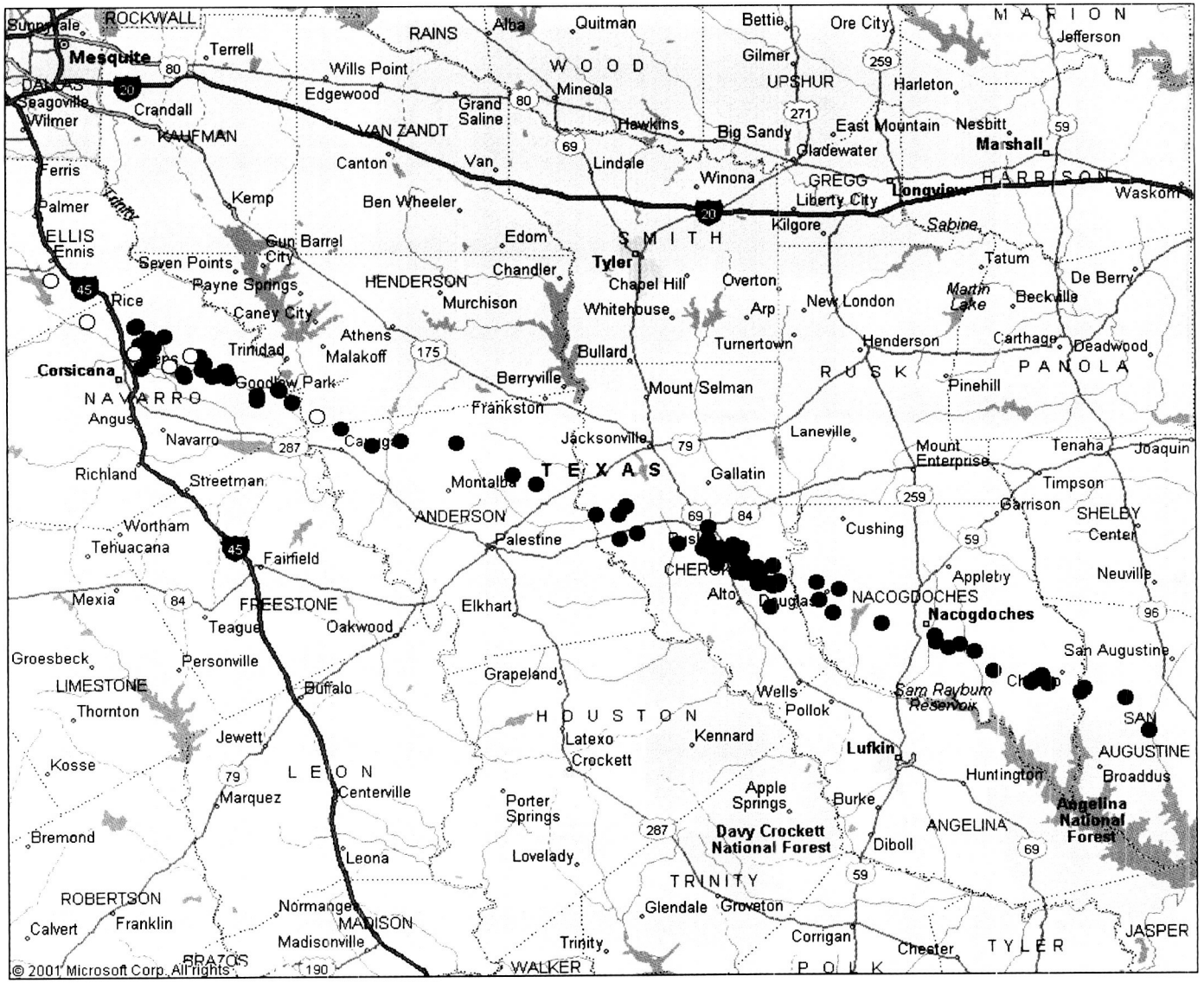
Spotlight/focus shifts to Left Wing Leading Edge damage

Location of each part found tracked on map - Trend emerges.

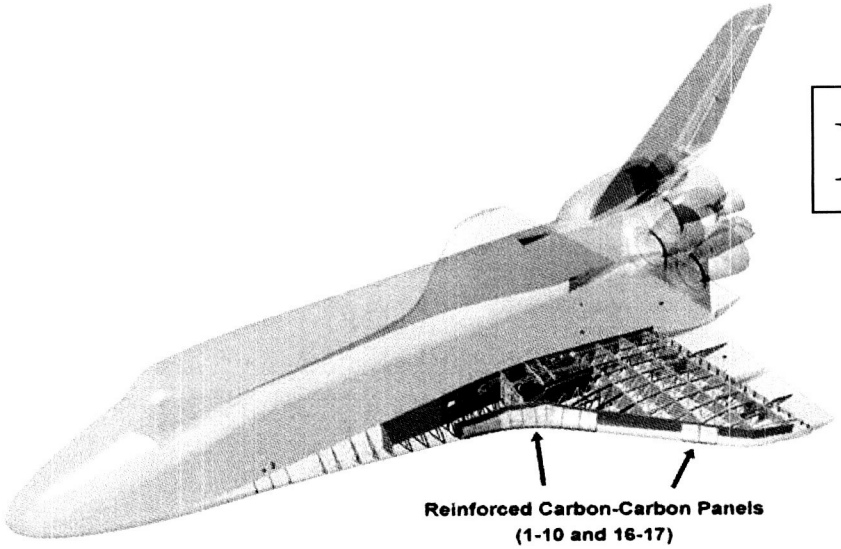
● Left Wing RCC

○ Left Wing Eroded RCC

● Right Wing RCC

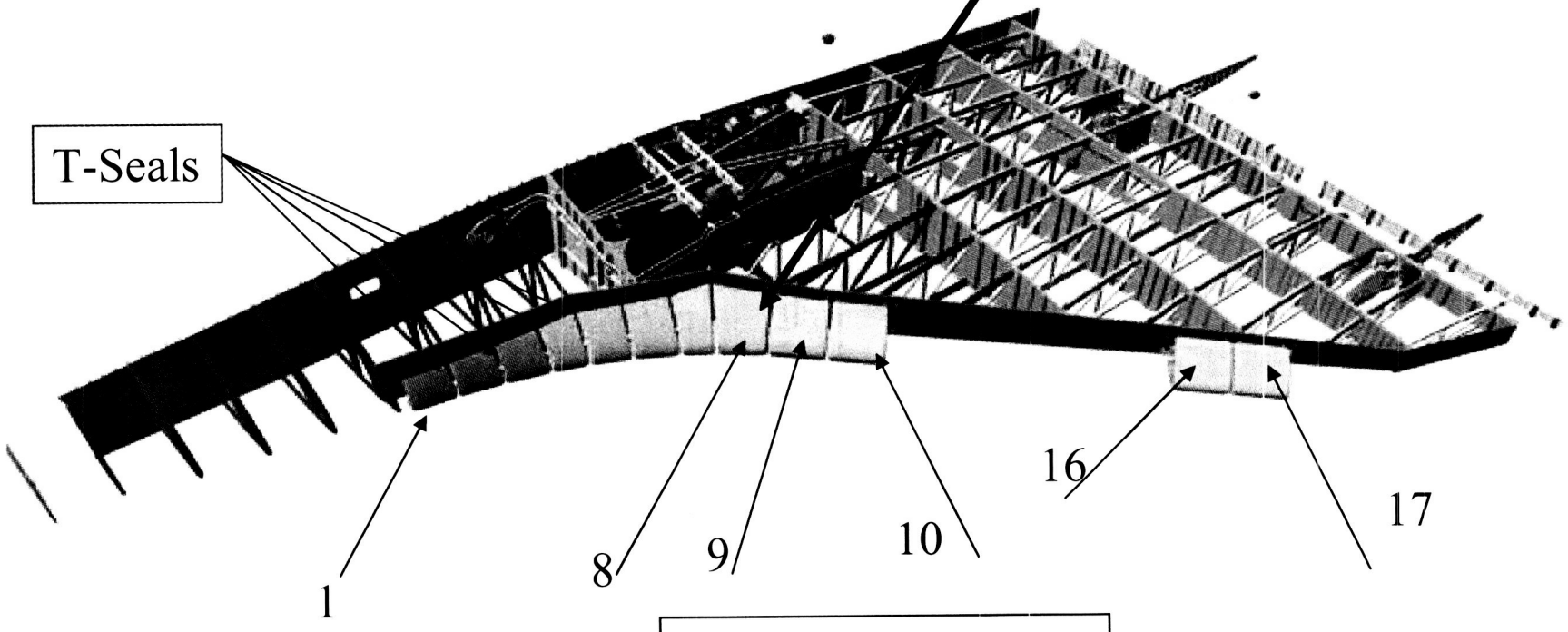


Left Wing Leading Edge



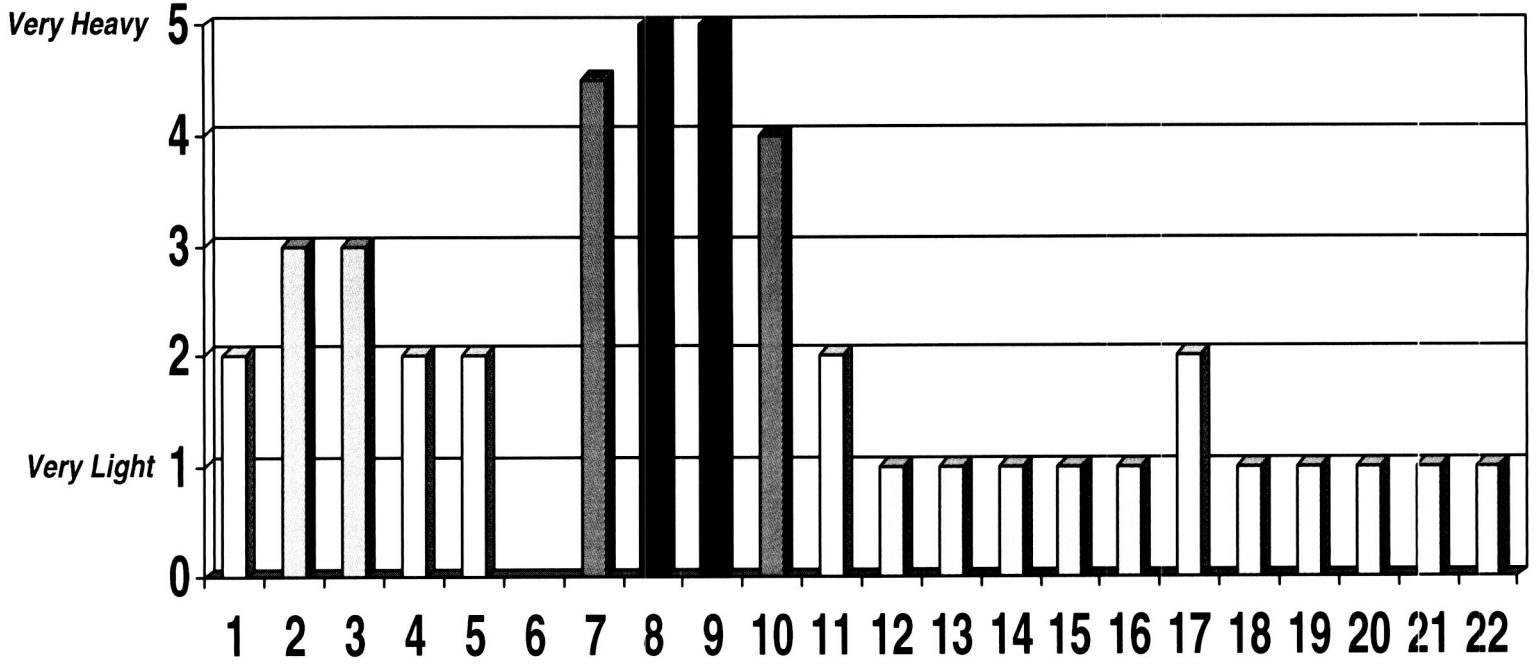
Highest Heating during Re-entry

T-Seals

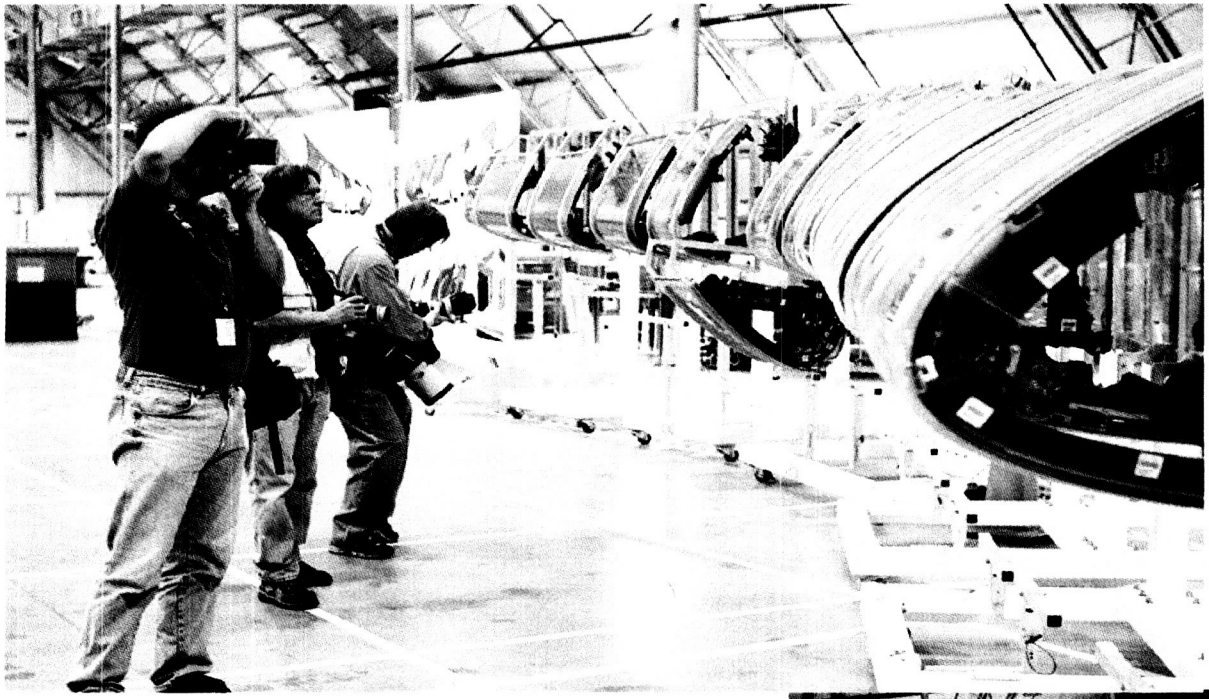


RCC Panel Numbers

Qualitative Slag Deposition Assessment "Very Light" to "Very Heavy"



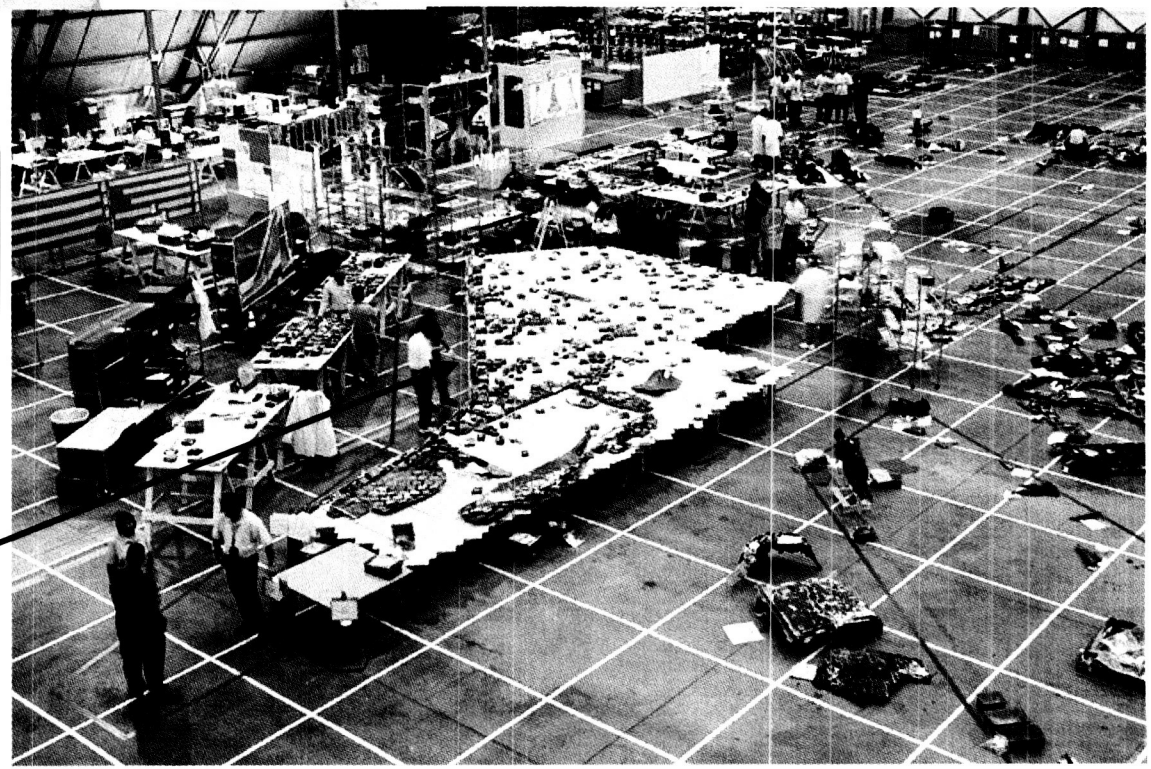
Distribution of "slag" deposition <u>volume</u> was centered around panels 8 & 9 on Left Wing Leading Edge RCC.

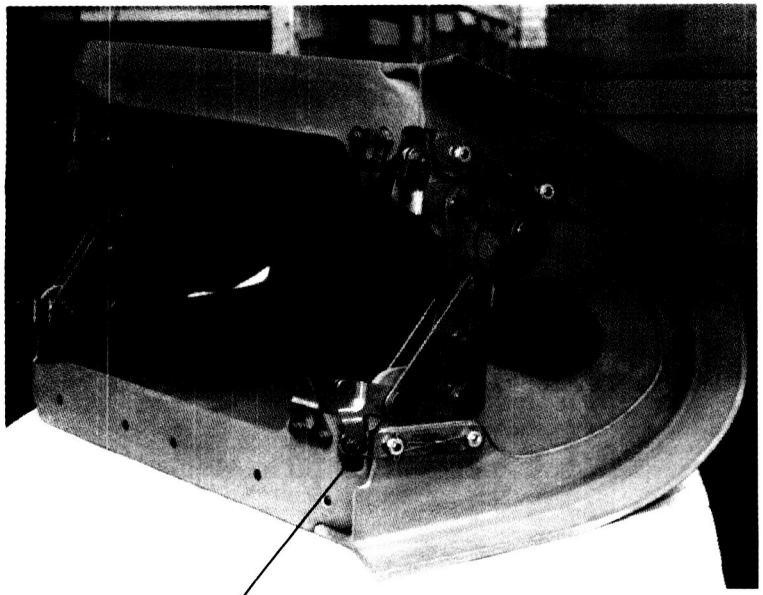


Left Wing Leading Edge
Final 3D Reconstruction

Left Wing Tile
Reconstruction

Leading Edge Tiles



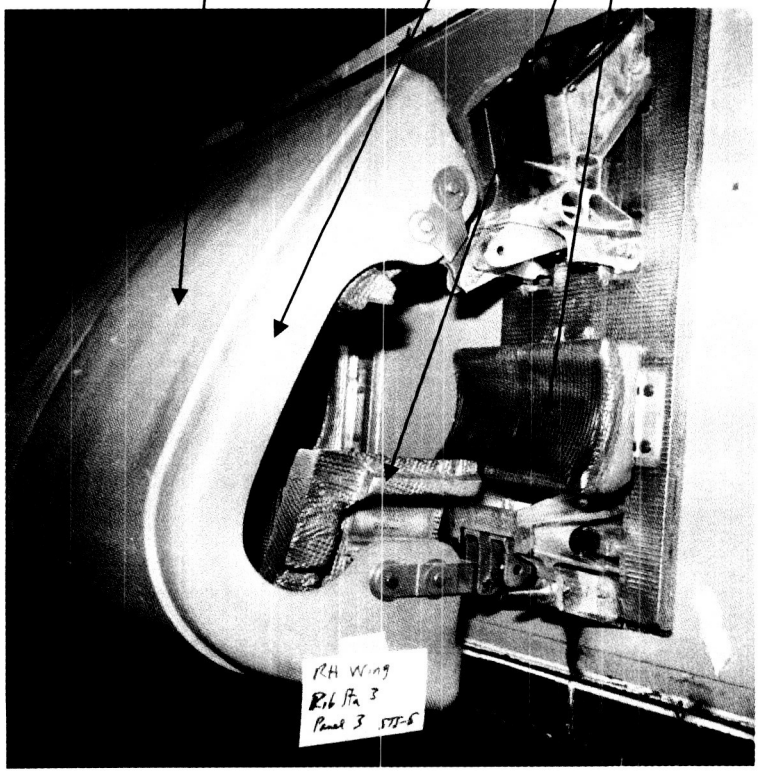


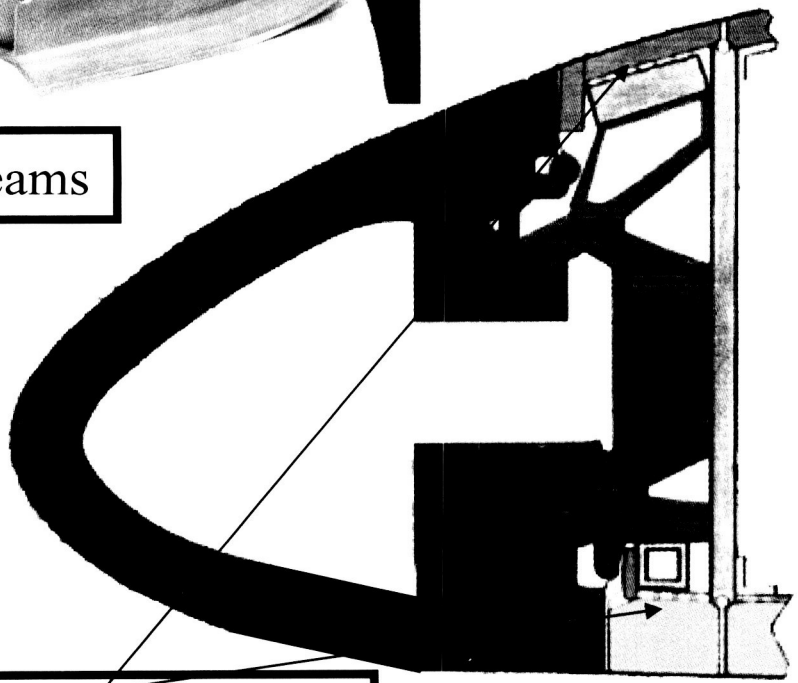
Spanner Beams

Dynaflex insulation – In601 foil with cerachrome fibers inside

RCC panel

RCC rib





Closeout/Carrier Panels and Tiles

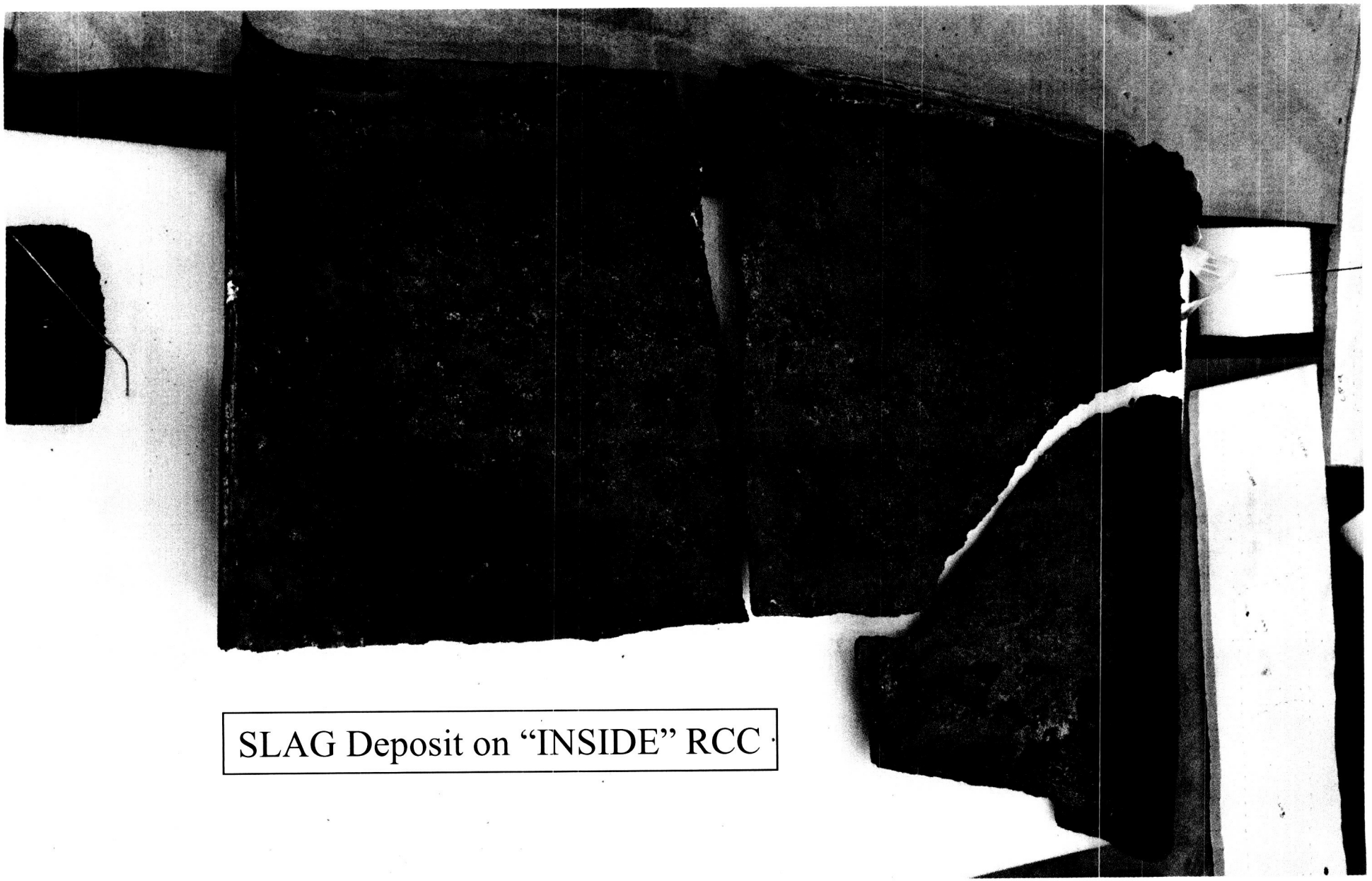
RCC
Aluminum
LI2200
LI900
Inconel-Dynaflex
Inconel 718
A-286 steel

Major Wing Leading Edge Materials and compositions

Insulators	IN601, Cerachrome	Ni-24Cr-14Fe-3Al-5Co-0.3Mn, Al2O3+SiO2+Cr2O3 (3200F)
Insulator Clip	Inconel 625	Max Ox resistance temp-1925F, Melting Point-2400F
RCC Clevis	Inconel 718	
Clevis Bracket	Inconel 625	Atomic: Ni-25Cr-3Fe-0.25Ti-0.5Al-5.6Mo-2.3Nb-Co-Mn
Insulator Strap	Inconel 718	
Fitting - Adjustable	A-286	
Spanner Beam	Inco 718	Max Ox resistance temp-1800F, Melting Point-2550F
Tee Clevis	Inco 718	Atomic Comp: Ni-21Cr-19Fe-1Ti-1Al-2Mo-3-Nb-5Co
Wick Plate	Inco X-750	
Shear Fitting/Wick	Inco 718	
Shear Pin/Ratchet	A-286	Max Ox resistance temp-1500F, Melting Point-2550F
Spar Attach Fitting	A-286	Atomic Comp: Fe-24Ni-16Cr-2.5Ti-0.7Al-0.8Mo-1Mn-0.33V
Spar	2024	MP-1200, Al-1.92Cu-1.71Mg-0.3Mn
Tiles	SiO2	SiO2
Gap Filler	Al2O3, Nextel	Al2O3
Bond Material	RTV, Nomex Felt	
Carrier Panel	2024 + 6061	
CP Fasteners	A286	

IN625, IN601, IN718, A286, 2024, Cerachrome, SiO2, 6061, Nextel Fabric, RTV

Example Debris, LH RCC 8



SLAG Deposit on "INSIDE" RCC

High Level Questions to M&P

Sample the slag deposits on RCC & Tiles to:

- Identify the location of breach in the wing leading edge.
- Identify the sequence of deposition/events
- Understand plasma flow direction and related thermal damage.

Analysis Techniques Arsenal

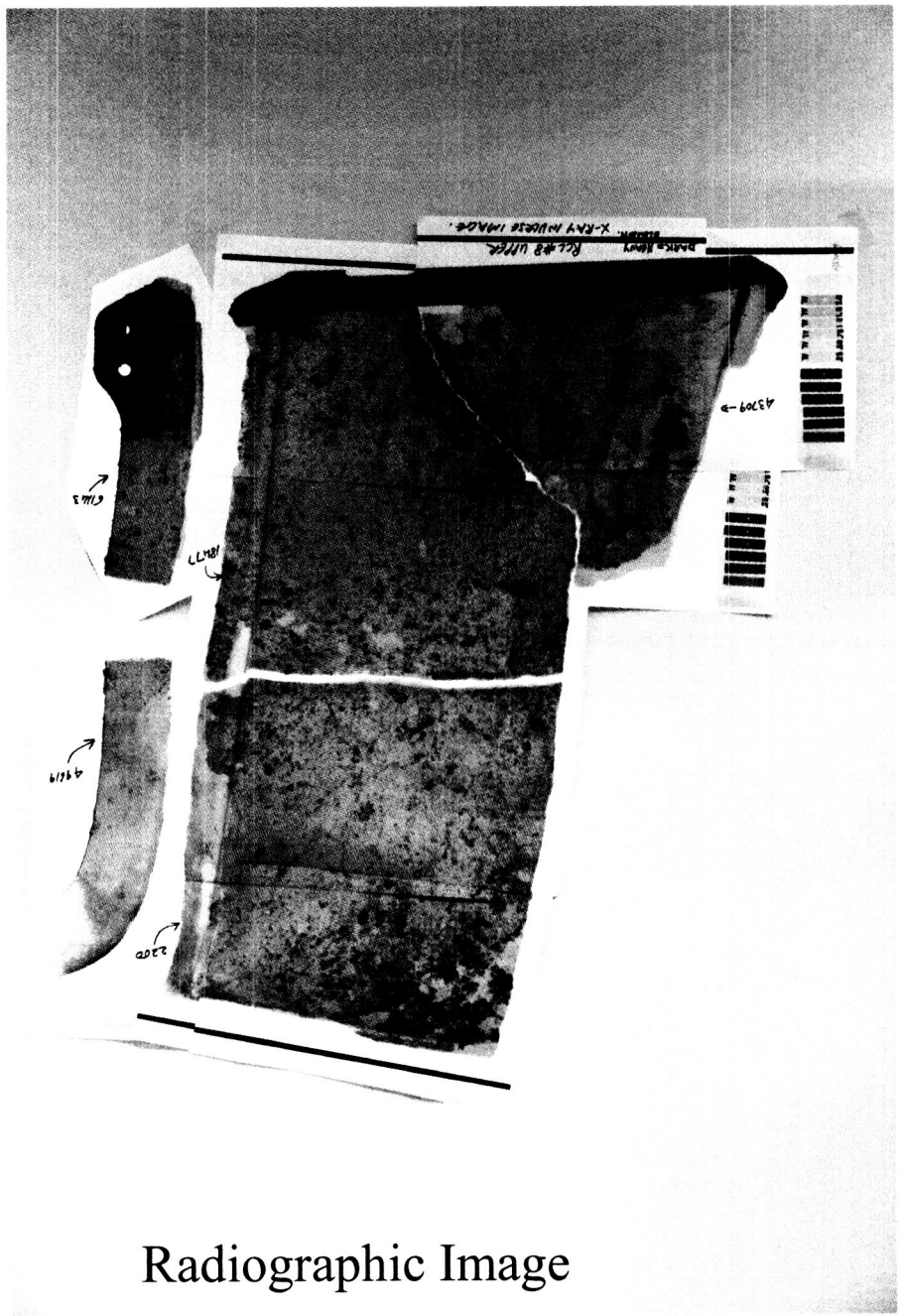
Analysis Technique	Purpose	Why/Advantages
Photography	Photo documentation	Documentation to maintain traceability
Scanning Electron Microscopy – SEM/EDS	Semi-quant elemental composition Back Scattered Imaging	Elements present, identify difference between top and bottom, X-ray mapping.
X-ray Diffraction - XRD	Identify compounds	Identify compounds of crystalline structure
Electron Microprobe	Identify elements	Determine exact composition
Fourier Transform Infra-Red - FTIR	Qualitative organic composition	If organic, aid in identification
ESCA/XPS	Identify inorganic & organic compounds	Aid in tracking of oxidation states, such al oxide; compound identification
Metallography + SEM	Layering of material	Composition through deposit layers
Inductively coupled plasma - ICP	Quantitative elemental composition	Elements present, Quantify bulk composition of sample
Radiography, CT, Ultrasonics	NDI and identification	See through the material, identify differences in materials, identify defects

Repeatability and Reproducibility of results emphasized

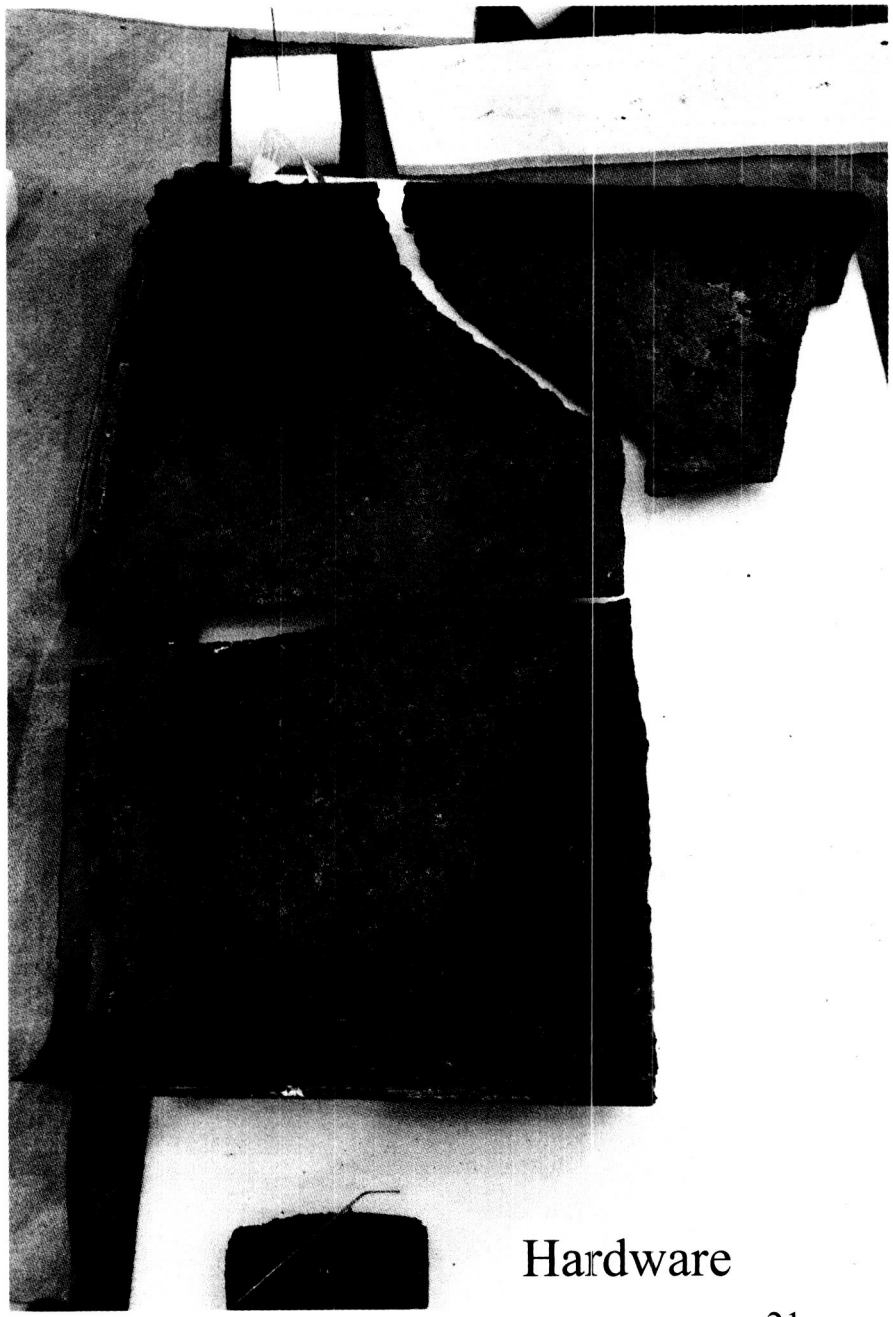
Analysis Approach Drivers

- **Strategically locate samples and reduce their number** - Radiography
- **Repeatability and Reproducibility** – take two samples of each feature.
- **Identify layering** – Through X-section
- **Identify elemental distribution** – SEM/EDS/X-ray Map, Back Scattered Imaging.
- **Identify compound formation** – X-ray diffraction and XPS
- **Quantify deposit chemistry** – Microprobe calibrated by standards

- **Relate the chemistry to Wing Leading edge source** - Accurate atomic ratios of specific elements present in slag obtained from Microprobe analysis identify the leading edge material source
- **Deposit Timeline** – Layering of wing leading edge material identifies the sequence of events
- **Global correlation of all data** – Multiple x-sections (located across multiple RCC panels) correlated with radiographic results to yield consistent layering information and thus the global sequence of events



Radiographic Image



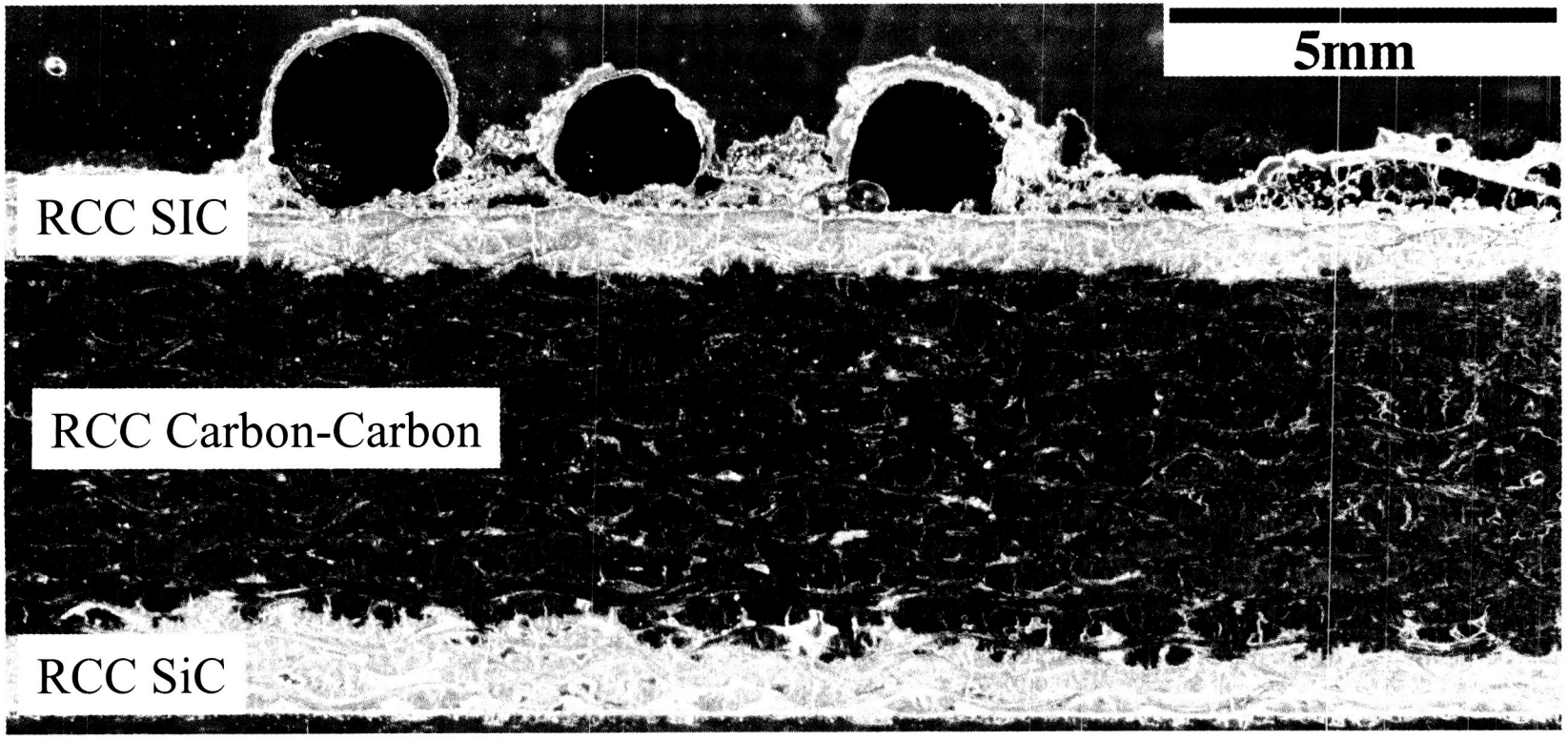
Hardware

Radiography of Slag Deposits on Left Wing RCC panel 8

Sample Location in Panel LH #8

Sample 3 X-section

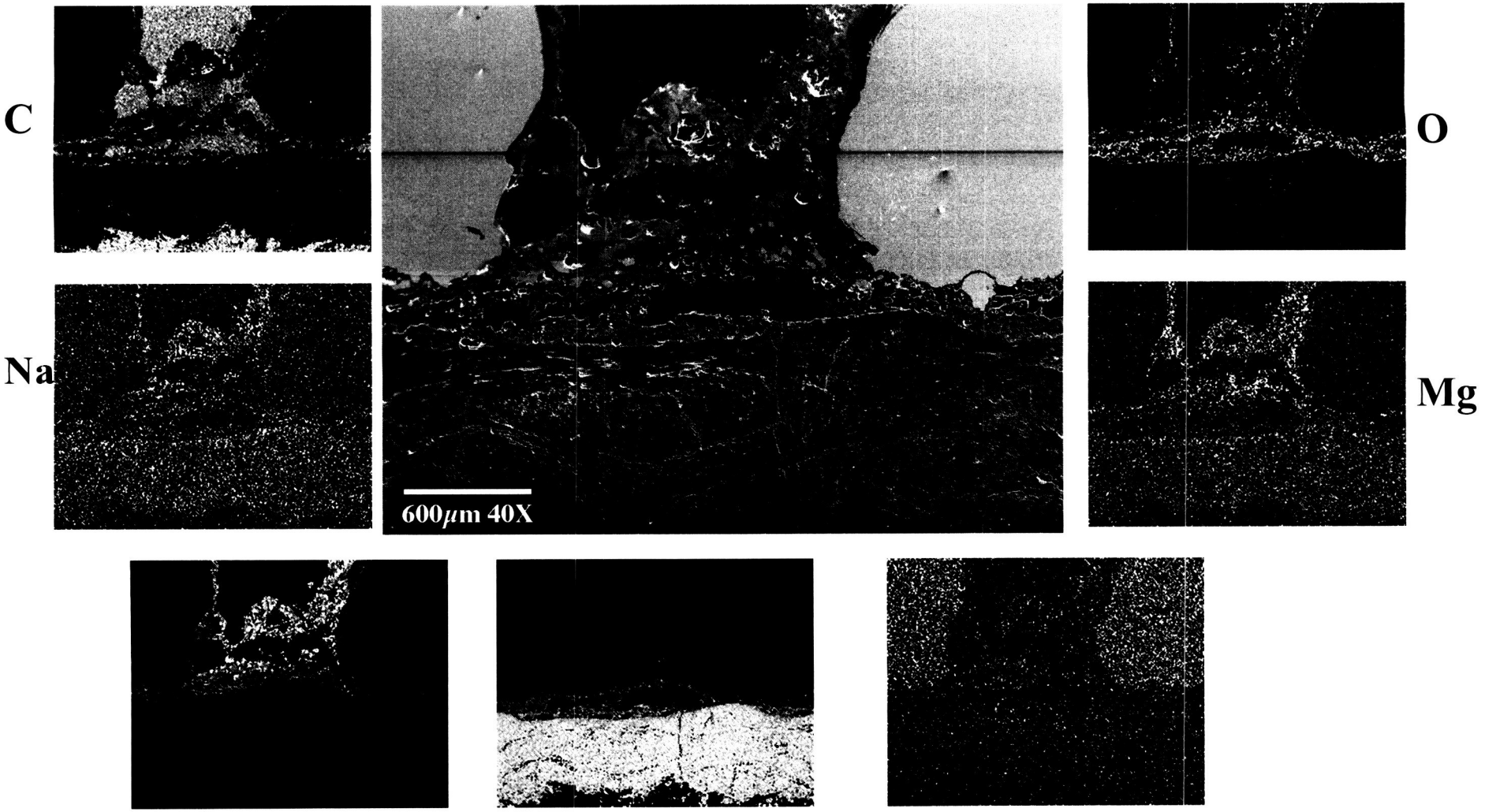
Inside Surface



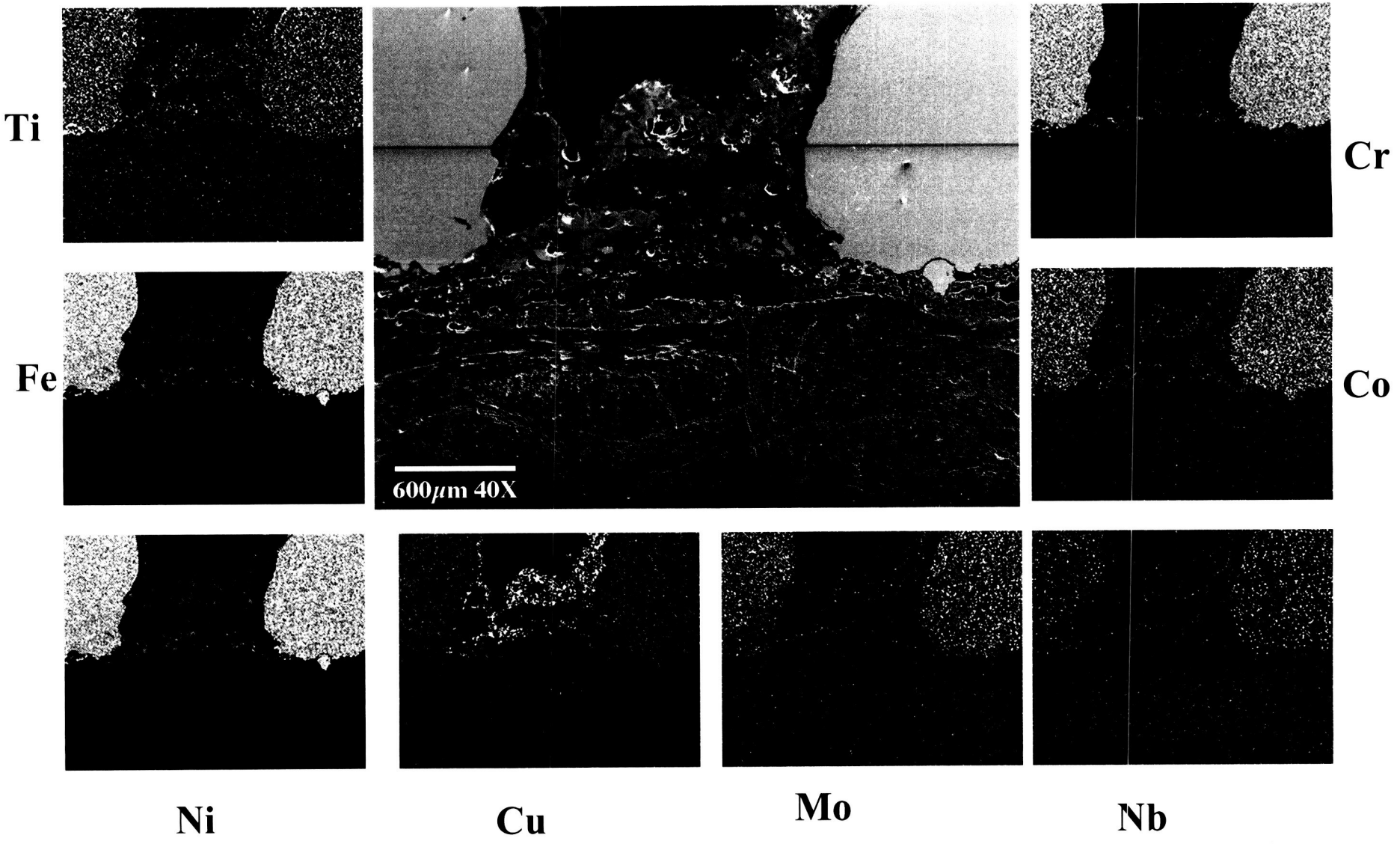
Outside Surface

Successful in cutting through the feature of interest guided by radiography

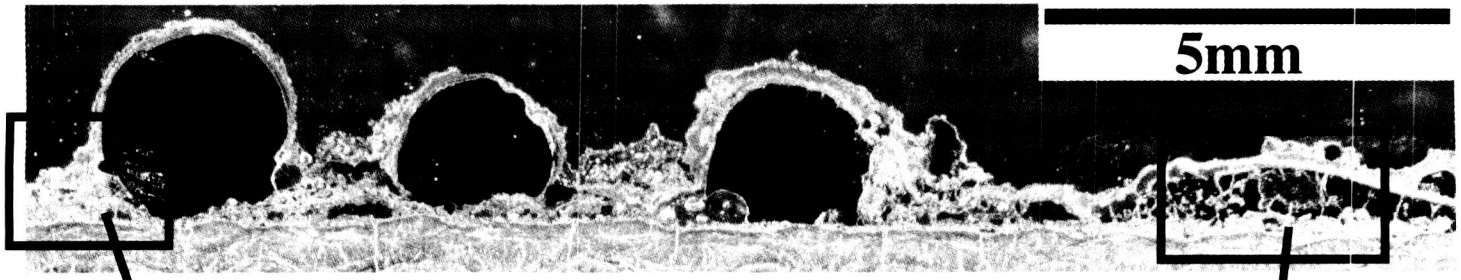
Elemental X-ray Dot Map - SEM

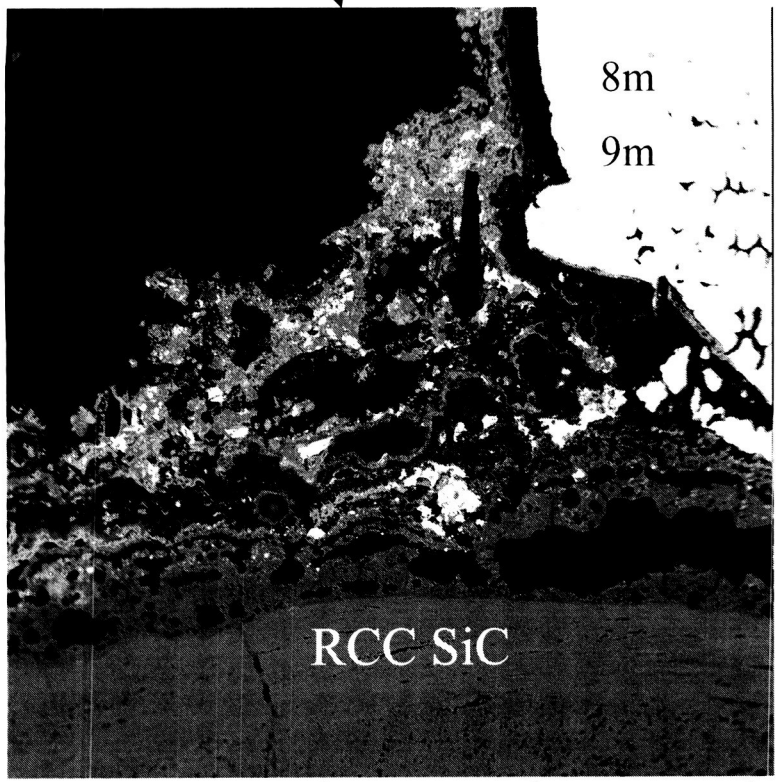


Elemental X-ray Dot Map - SEM

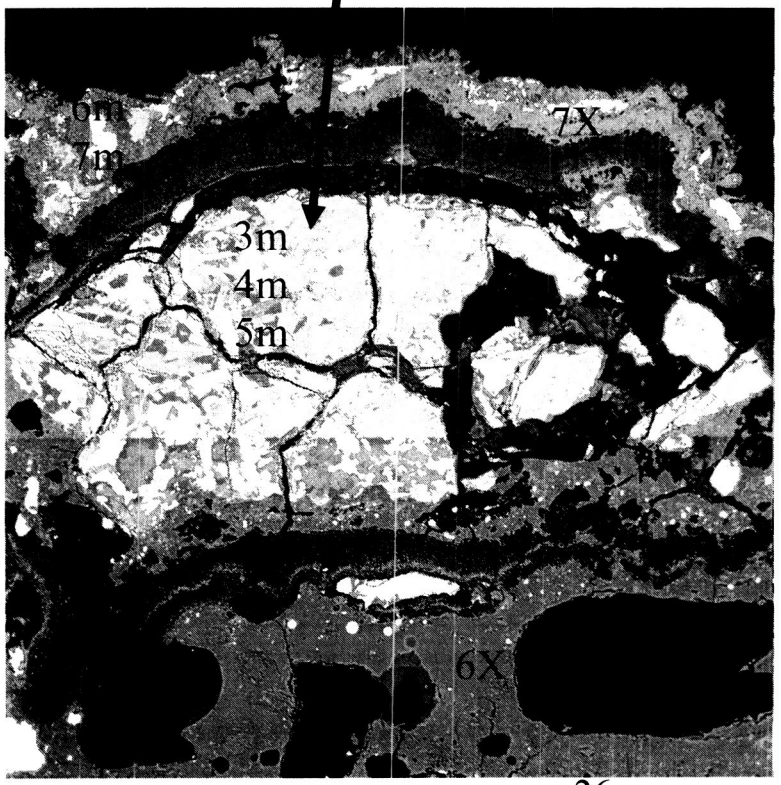


Backscattered Electron Imaging and Microprobe chemical analysis





M = Metallic
X = Oxide



BackScattered Images showing contrast between Metals and Oxides

Quantitative Chemical Analysis in Atomic %

Point	Cr	Ti	Mo	Nb	Ni	Fe	Co	Cu	Si	Al	Mg			
3m	0.44	0.02	0.01	0.02	29.9	2.27	0.1	4.98	3.12	59.14	0			
4m	5	0.01	0.21	0.03	7.34	13.81	0.13	0.53	6.25	66.68	0.01			
5m	4.37	0.02	0.13	0.03	2.48	10.87	0.08	1.23	10.95	69.84	0			
6m	0.14	0	0	0	12.21	4.73	0.13	1.19	1.7	79.9	0			
7m	0.13	0	0	0	26.67	0.57	0.04	10.74	0.61	61.23	0			
8m	18.53	0.16	1.19	0.52	46.65	26.91	0.39	0.14	2.25	3.26	0			
9m	1.79	9.41	2.11	82.74	2.5	1.4	0.03	0.03	0	0	0			
Point	Na	Mg	Al	Si	Ni	Fe	Co	Cu	Cr	Ti	Ca	Mo	Nb	O
5X	0.06	0.03	22.87	1.65	0.86	0.57	0	0.14	0.63	0	0.01	0	0	73.19
6X	0.16	0.21	3.95	28.57	0.1	0.14	0	0	0.29	0.01	0.05	0.01	0	66.51
7X	0.01	0.37	40.49	13.15	0.24	0.12	0	0.4	0.07	0.01	0.02	0	0	45.12

Interpretation Criteria - Examples

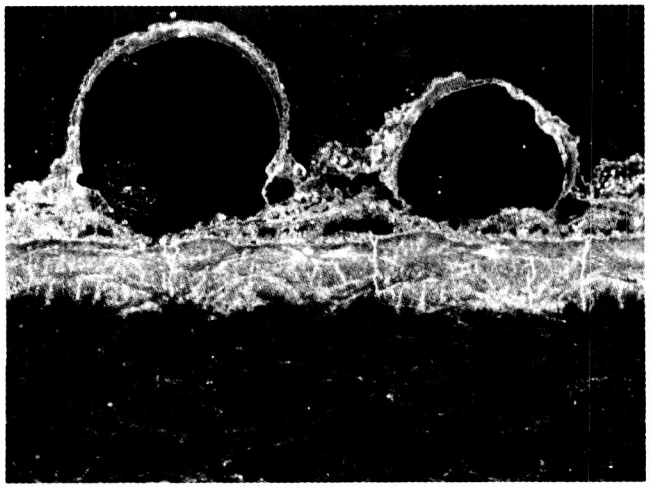
- **How to identify specific alloys in the deposit?**
  - A286 or IN601, IN718, IN625 can be distinguished based on (Ni/Fe) ratio and evidence and amounts of Mo, Nb, Co and Ti.
  - 2024 can be identified by presence of metallic Al + Cu, Al_2O_3 + Cu.
- **How to identify Cerachrome in deposit?**
  - Cerachrome is approximately 43%$Al_2O_3$53%$SiO_2$3%Cr_2O_3.
  - It can be identified from a combination of back-scattered imaging, color, x-ray diffraction and presence and quantification of Al, Si, O, & Cr.
- **How to identify SiO2 from Tile?**
  - SiO2 from tile will not have with other elements as in cerachrome. It could still pick up a coating of alumina then morphological features will be used to distinguish.

Interpretation

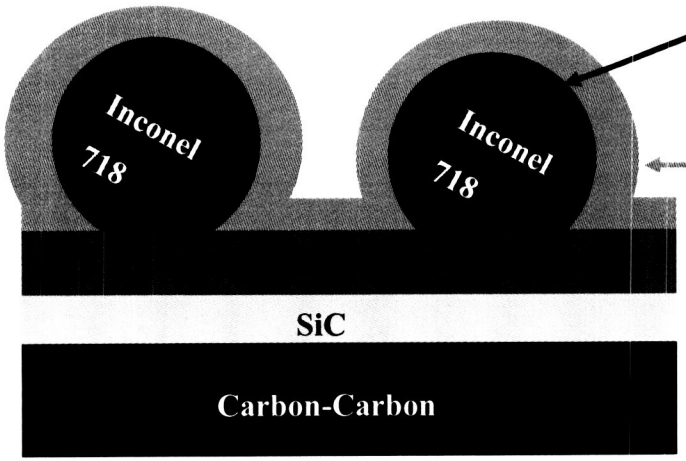
Point	Analysis Notes	Source Identification (Major component in Bold)
3m	Al+Ni, some Fe+Cr+Ti+Cu, minimal Mo+Nb	**2024Al**, **IN601**, **SiC**
4m	Al+Fe, Some Ni+Cr+Ti+Cu, minimal Mo+Nb	**2024Al**, **IN601**, **SiC**
5m	Al+Si+Fe, some Fe+Cr+Ti+Cu, minimal Mo+Nb	**2024Al**, **IN601**, **SiC**
6m	Al+Ni, some Fe+Cr+Si, no Ti+Mo+Nb	**2024Al**, **IN601**, **SiC**
7m	Al+Ni+Cu, Some Fe+Cr+Si, no Ti+Mo+Nb	**2024Al**, **IN601**, **SiC**
8m	Ni+Fe+Cr, some Si+Al+Ti+Mo+Nb, Ni/Fe=1.7	**IN718**, **2024Al**, **SiC**
9m	Nb+Ti, some Ni+Fe+Cr	**IN718**, **2024Al**, **SiC**
Point		
5X	Alumina+Cu, Some Silica +Cr	**2024Al**, **Cerachrome**
6X	Silica+Na, Some alumina+Cr	**Type A coating**
7X	Alumina+Cu+Silica+Na+Cr, some metallic Al	**2024Al**, **Cerachrome**, **Type A**

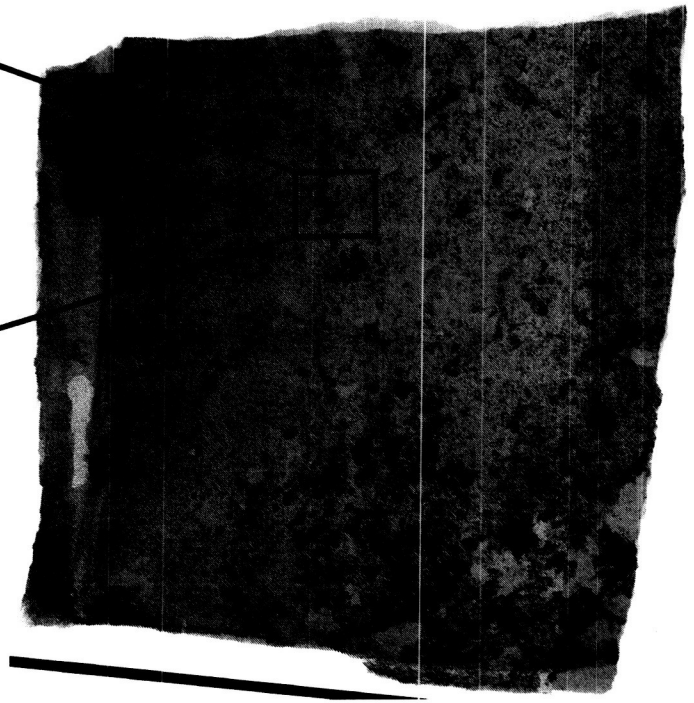
No A286 observed consistently

Left Wing RCC #8 - Slag Feature Spheroids

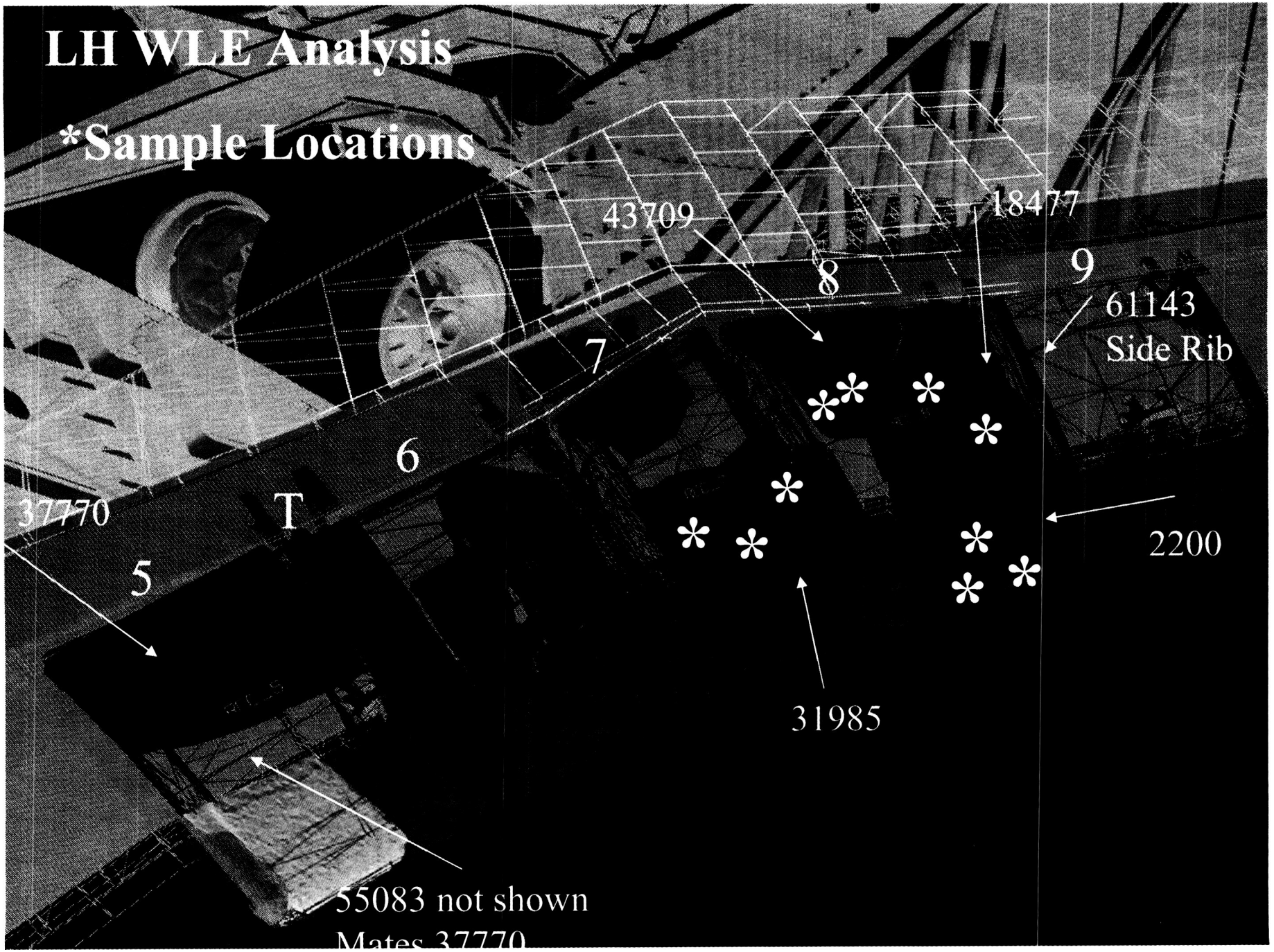


Slag Item 2200, Sample 6C1

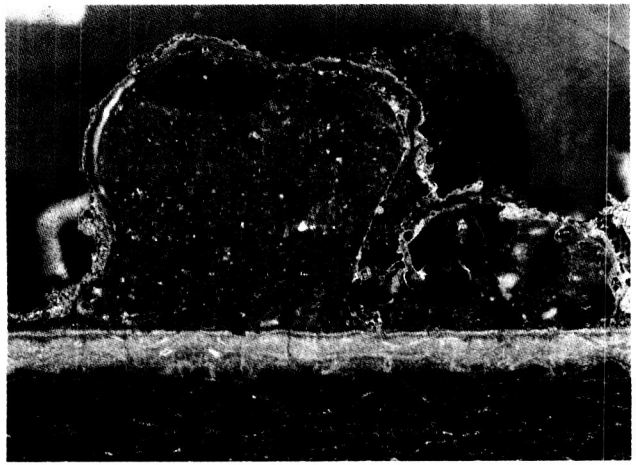




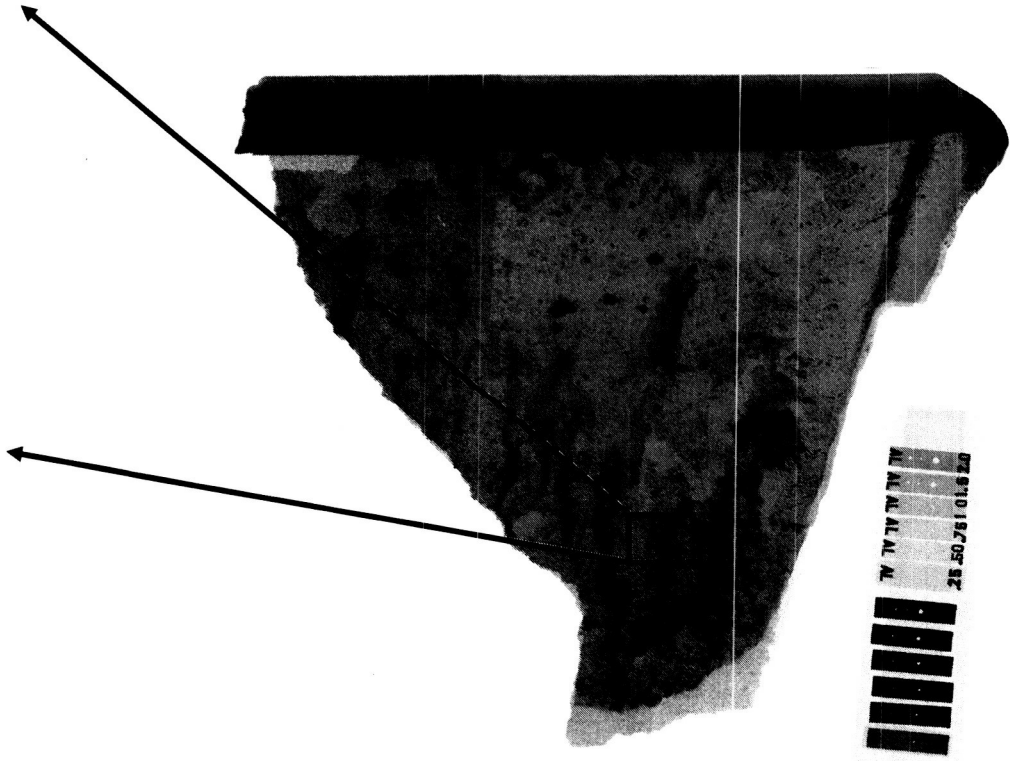
Radiograph of Slag Item 2200


LH WLE Analysis
*Sample Locations
43709
18477
8
9
61143
Side Rib
7
6
37770
T
5
2200
31985
55083 not shown
Mates 37770

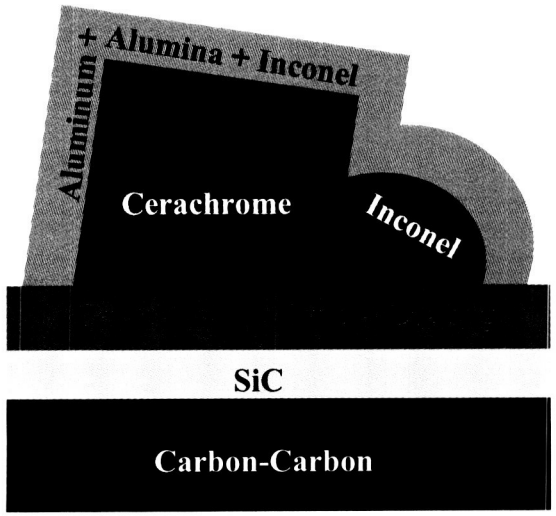
Left Wing RCC #8 - Slag Feature Thick Tear Shaped



Slag Item 43709, Sample 2A1

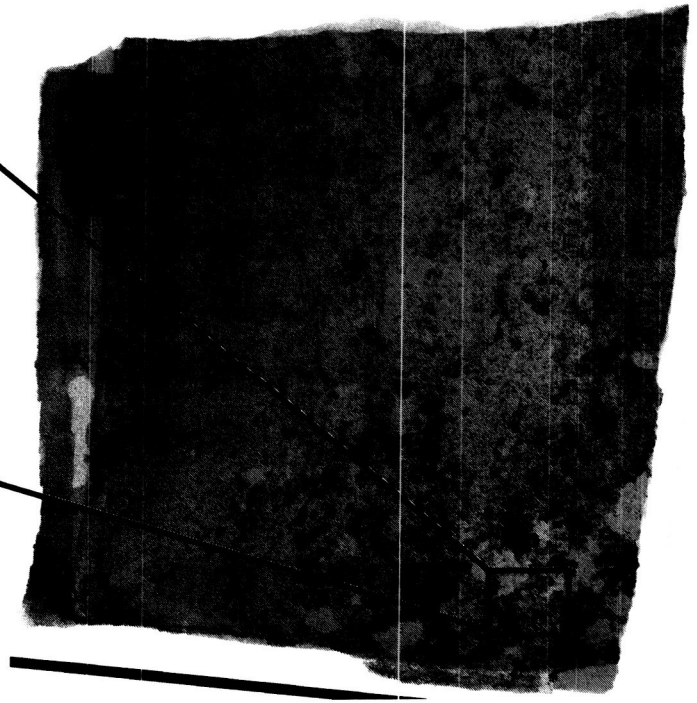


Radiograph of Item 43709

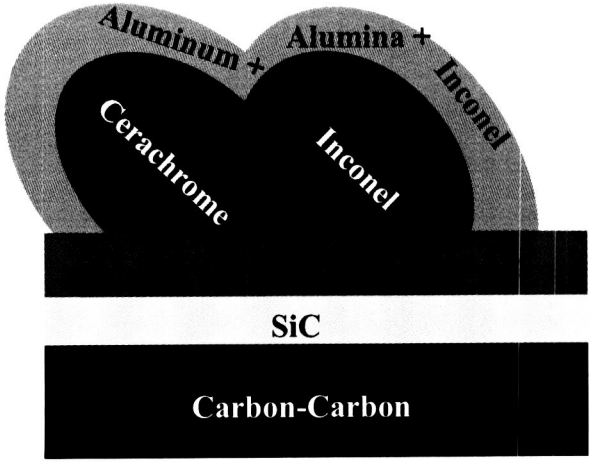


Left Wing RCC #8 - Slag Feature Thick Globules

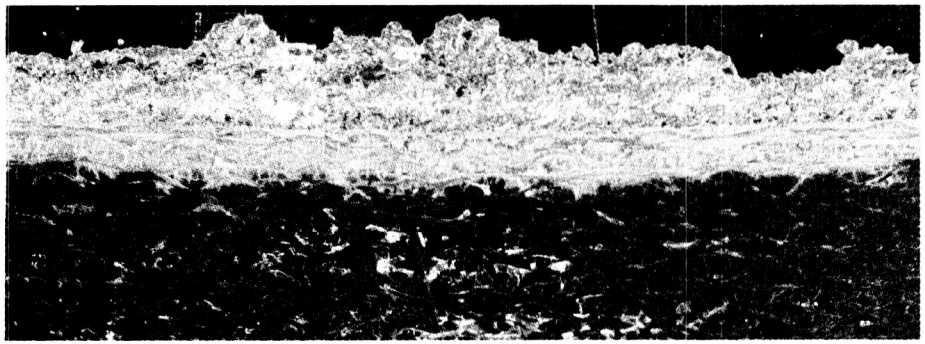
Slag Item 2200, Sample 6A1



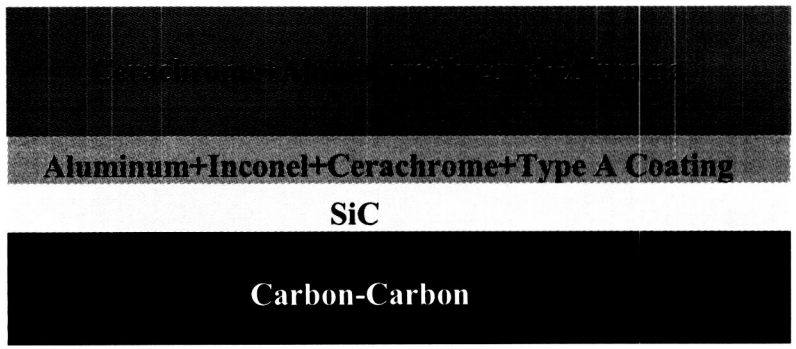
Radiograph of Item 2200

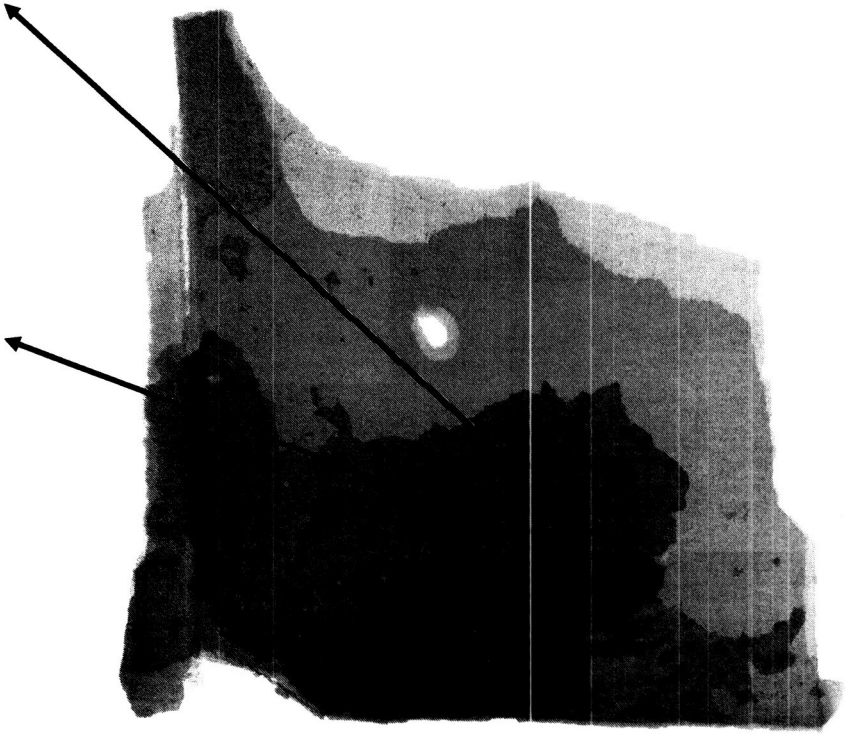


Right Wing RCC #8 - Slag Feature
Uniform Deposit



Slag Item 16523, Sample 4A1





Radiograph of Item 16523

RCC Slag Significant Findings
LH RCC #8

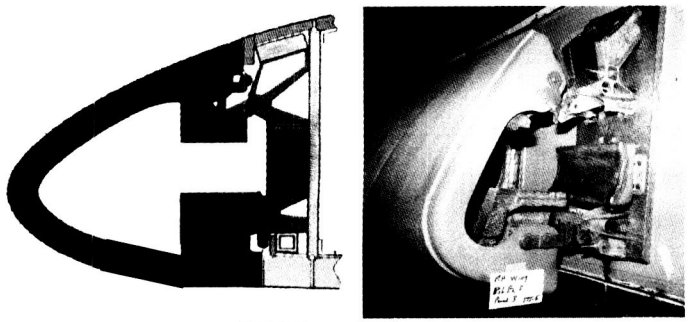
- Cerachrome + Inconel in first deposited layers
  - ➢ Melting of spanner/foil/fittings + Insulator
- Aluminum deposition secondary event

Slag layering suggests plasma impingement location

Slag distribution & shape suggests plasma flow direction and deposition duration

- No indication of stainless steel spar fittings (A286) in slag
  - ➢ Breach location away from spar fittings
- Large amounts of melted ceramic cerachrome insulator
  - ➢ High temperature >3200°F

Reconstructed View
LH CPanel 9 Tiles, lower



Horse Collar Fabric Deposit

C P 8

Insert

Tile Slumping

57754 # 22571 # 50338 # 16692

Molten Slag on Tile

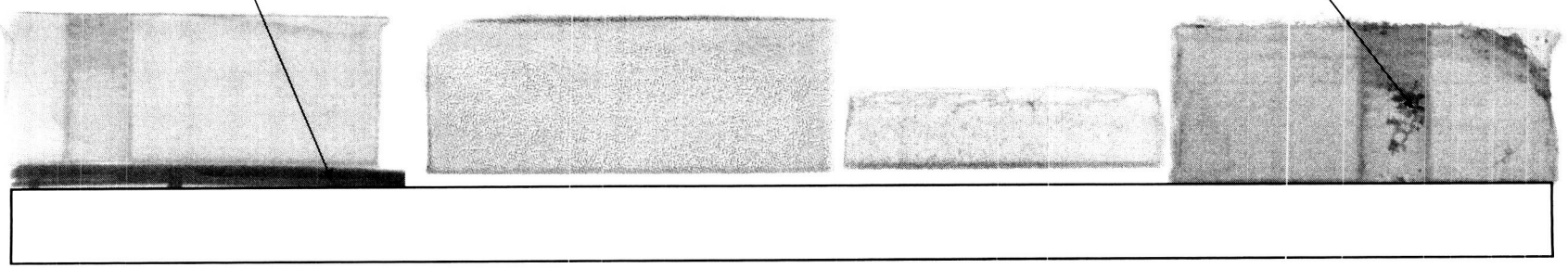
Spar

RCC

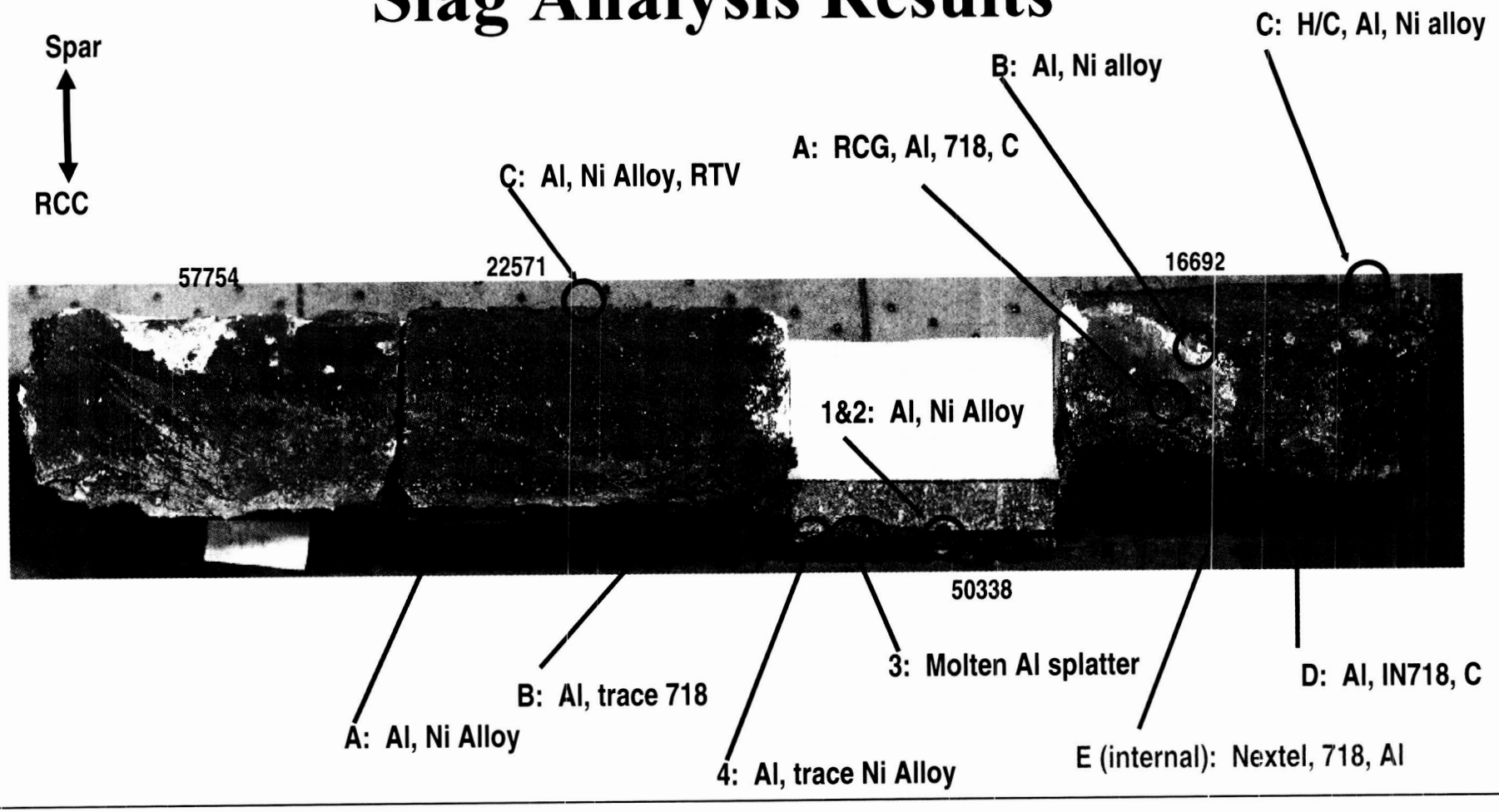
Realtime X-ray, Sidewall View

Carrier Panel

High-Z material

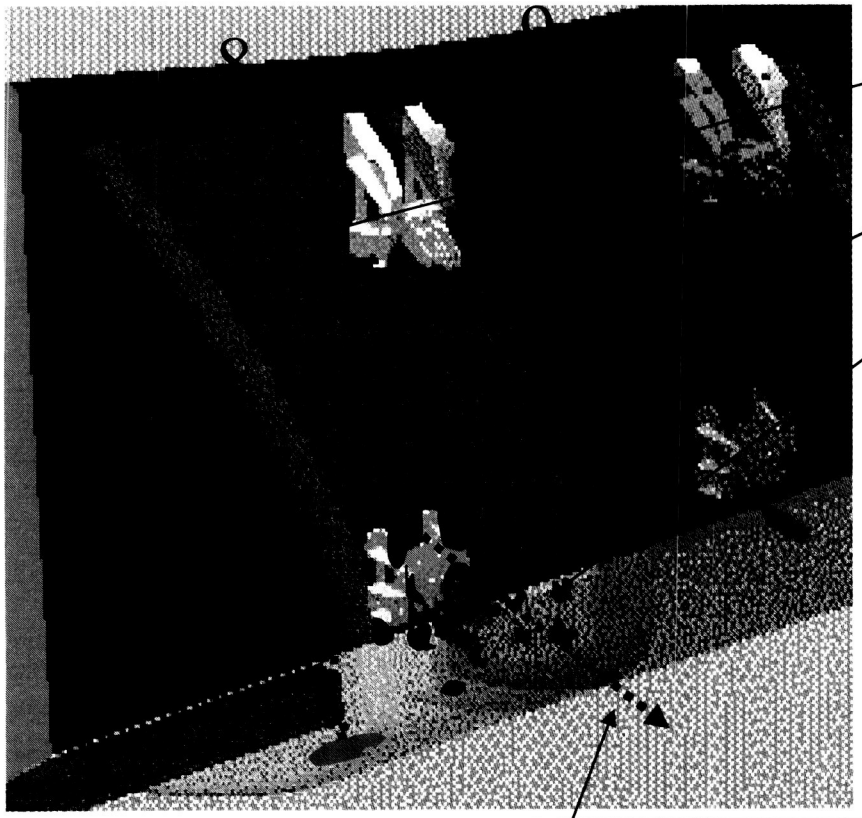


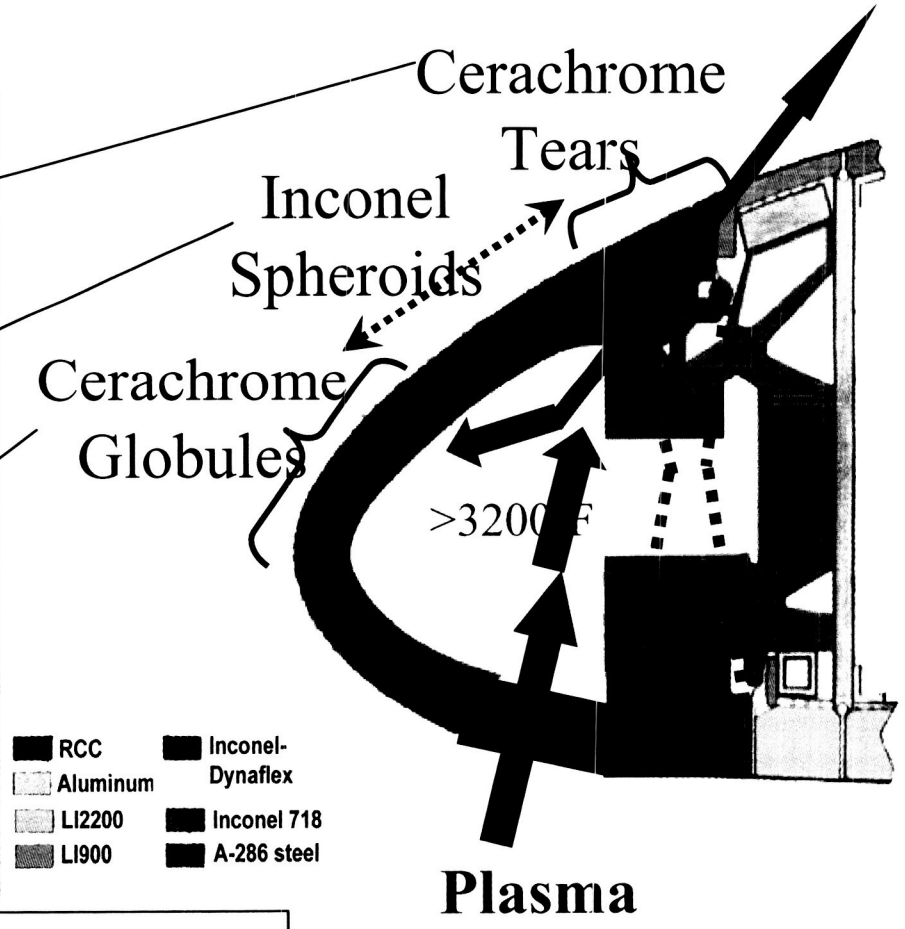
Reconstructed View
LH Carrier Panel 9 Tiles, Lower
Slag Analysis Results



These findings suggest flow of material from inside the RCC out through the upper and lower CP locations.

Proposed Breach Location & Plasma Flow Based On Slag Results





RCC
Aluminum
LI2200
LI900
Inconel-Dynaflex
Inconel 718
A-286 steel

Flow Exiting through RCC 8 on to lower Carrier Panel 9 tiles